



## **FINAL REPORT**

**October 2001**

# **Base Hydrolysis Process for the Destruction of Energetic Materials**

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## Executive Summary

The Program Manager, Assembled Chemical Weapons Assessment (PM ACWA) was formed by Public Law 104-208, Sec. 8065 to study alternatives to the baseline incineration process for the demilitarization of assembled chemical weapons, and that at least two alternatives to the baseline incineration process be identified and demonstrated. The information generated under the PM ACWA program will be used to support a technology decision for the Pueblo, Colorado and Blue Grass, Kentucky Chemical Agent Disposal Facilities.

In FY 99, PM ACWA decided to conduct additional work to optimize the hydrolysis process for energetic materials, an intermediate processing step used to de-energize the energetics recovered from chemical munitions during the disassembly operation.

Tank-Automotive and Armaments Command - Armament Research, Development and Engineering Center was tasked by PM ACWA to execute an Engineering Scale Test of the base hydrolysis process. The objective of this program was to: determine optimum operating parameters to support scale-up of the hydrolysis process, define a hydrolysis process that is safe and environmentally compliant; and address issues regarding full-scale hydrolysis of energetics identified by the National Research Council (NRC) reviewing technical progress on the ACWA program.

Commissioning of the energetics hydrolysis system was successfully accomplished at Holston Army Ammunition Plant on 14 December 00 with the first trial run with Composition B explosive. The test and evaluation program was completed in April 01.

The results of the testing indicate that the base hydrolysis process for energetics is robust, reliable and flexible. The process will easily achieved Destruction Rate Efficiency (DRE) ranging from 99.75% to 100% versus a goal of 99.999%. Where the 99.999% goal was not achieved (the sampling and analysis procedure may have contributed to the lower than desired DRE), the hydrolysate could be safely processed by the final treatment step. The concerns identified by the NRC have been satisfactorily addressed; i.e., the by-products of full-scale processing of energetics are relatively benign.

The formation of Picric Acid as a by-product of energetics hydrolysis is not considered a problem. Picric Acid was only detected at very low levels in the mid-run analyses for Tetrytol and was detected at even lower levels in the end of run analyses. This conclusion is supported by the bench-scale work performed by LANL that showed no Picric Acid present in the hydrolysate.

Processing energetic mixtures presented no problems and can be safely performed with the process. Los Alamos National Laboratory performed substantial bench-scale testing to support this effort and expand the database for the hydrolysis of energetics.

Two processing concerns were identified during the program: the handling of the rayon bags containing the M1 propellant charge, and the handling of the cotton threads used to bundle the M8 sheet propellant. Both warrant further study.

The full-scale system performed satisfactorily from an equipment standpoint. The only problem encountered was the feeding of the dry energetics using a loss-in-weight feeder. There were several improvements identified that were not implemented because of the severe schedule; and for the most part, these improvements were directed at improving data collection and not to address processing deficiencies.

The energetic hydrolysis system was successfully demonstrated on a pilot-scale and is recommended for inclusion in the design package for the Pueblo and Blue Grass Chemical Agent Disposal Facilities.

## **ACKNOWLEDGEMENTS**

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And congratulations for a job well done to Messrs. Andrew Wilson, and Mike Ervin, as well as all the other members of the RONA Project Team at Holston AAP,

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## **1.0 BACKGROUND**

PM ACWA was formed as a result of Public Law 104-208, Sec. 8065 that mandates a study of alternatives to the baseline incineration process for the demilitarization of assembled chemical weapons. At least two technologies must be demonstrated that will address all aspects of demilitarization of all components of each of the chemical weapons in the stockpile. The chemical weapon storage sites supported by the ACWA program are located at Pueblo Army Ammunition Depot (AAD), Pueblo, Colorado and Blue Grass AAD, Lexington, Kentucky.

### **1.1 Candidate Technologies.**

PM ACWA identified 6 candidate technologies that warranted further evaluation. Three of the technologies were evaluated on a bench-scale in FY 99:

- Bio-treatment (combined hydrolysates)
- Plasma arc
- Super critical water oxidation (SCWO)

Upon completion of the evaluation of these candidate technologies, the stakeholders ((public interests groups including Green Peace, the Sierra Club, local and state government representatives, subject matter experts, etc.) successfully petitioned Congress to provide additional funding to evaluate the remaining three candidate technologies: (see Appendix A for acronyms and abbreviations.)

- Solvated electron technology (SET)
- Gas phase chemical reduction (GPCR)
- SILVER II technology

These evaluations were conducted in the FY 00 and FY 01 time frame. The PM decided to conduct additional work to characterization and optimize the base hydrolysis process for energetic materials based on comments and recommendations received from the National Research Council (NRC) (Appendix B reviews the NRC concerns) regarding the base hydrolysis process for energetics (the NRC is independently reviewing the results of the ACWA program). Base hydrolysis is an intermediate process step used to de-energize the energetic materials (explosives and propellants) recovered from the chemical munitions during the disassembly operation. The hydrolysate produced during the hydrolysis of the energetics is sent to a final destruction process.

### **1.2 Energetics Hydrolysis System.**

TACOM-ARDEC was tasked by PM ACWA to conduct engineering scale testing (EST) with a pilot-scale hydrolysis system capable of processing all energetics (explosives and propellants) found in the chemical weapons stockpiled at the Pueblo and Blue Grass AADs. The system was to be full-scale capable of processing up to 500-pounds per hour of energetics.

#### **1.2.1 EST Energetics Hydrolysis System Program Team:**

The government team was comprised of government personnel from TACOM-ARDEC (technical managers of the program), Holston Army Ammunition Plant (AAP) (installation site for the energetics hydrolysis system), Radford AAP (manufacturing site for M28 surrogate propellant), and the Naval Surface Weapons Center. The contractors and OGAs supporting the EST effort included Royal Ordnance North America (operating contractor of Holston AAP), Alliant Techsystems (operating contractor for Radford AAP), IPS, Inc., Pfaudler, Inc., Pantex, Inc., and Los Alamos National Laboratory (LANL). Sample collection was overseen by A.D. Little, Inc. with TRC Inc. providing and manning the off-gas sampling system that was interfaced to the reactor.

## 2.0 PROGRAM OBJECTIVE

The objective of the TACOM-ARDEC Energetics Hydrolysis System EST program is to:

- Address concerns identified by the National Research Council (NRC) and processing issues that surfaced at Radford AAP and PANTEX, Inc. during the manufacturing of the various hydrolysates used to support the previous demonstration testing
- Determine the optimum process operating parameters to support scale-up of the hydrolysis process and the definitization of the Engineering Design Package (EDP) for the pilot phase for the Pueblo Chemical Agent Disposal Facility scheduled in August 2001 and for the Blue Grass Chemical Agent Disposal Facility scheduled in August 2002.
- Define a hydrolysis process that is safe and environmentally compliant, and that will efficiently produce hydrolysates of energetic materials recovered from the various chemical munitions during the disassembly process.
- Produce hydrolysates that will be ready for post-treatment processing using such technologies as SCWO, bioreactor, etc.

The hydrolysis process defined under this program will provide the flexibility to process the full range of material conditions that may be encountered with the recovered energetics, and to produce hydrolysates that conform to the material stream requirements dictated by final post treatment process.

### 2.1 Energetics Hydrolysis Pilot Plant Layout.

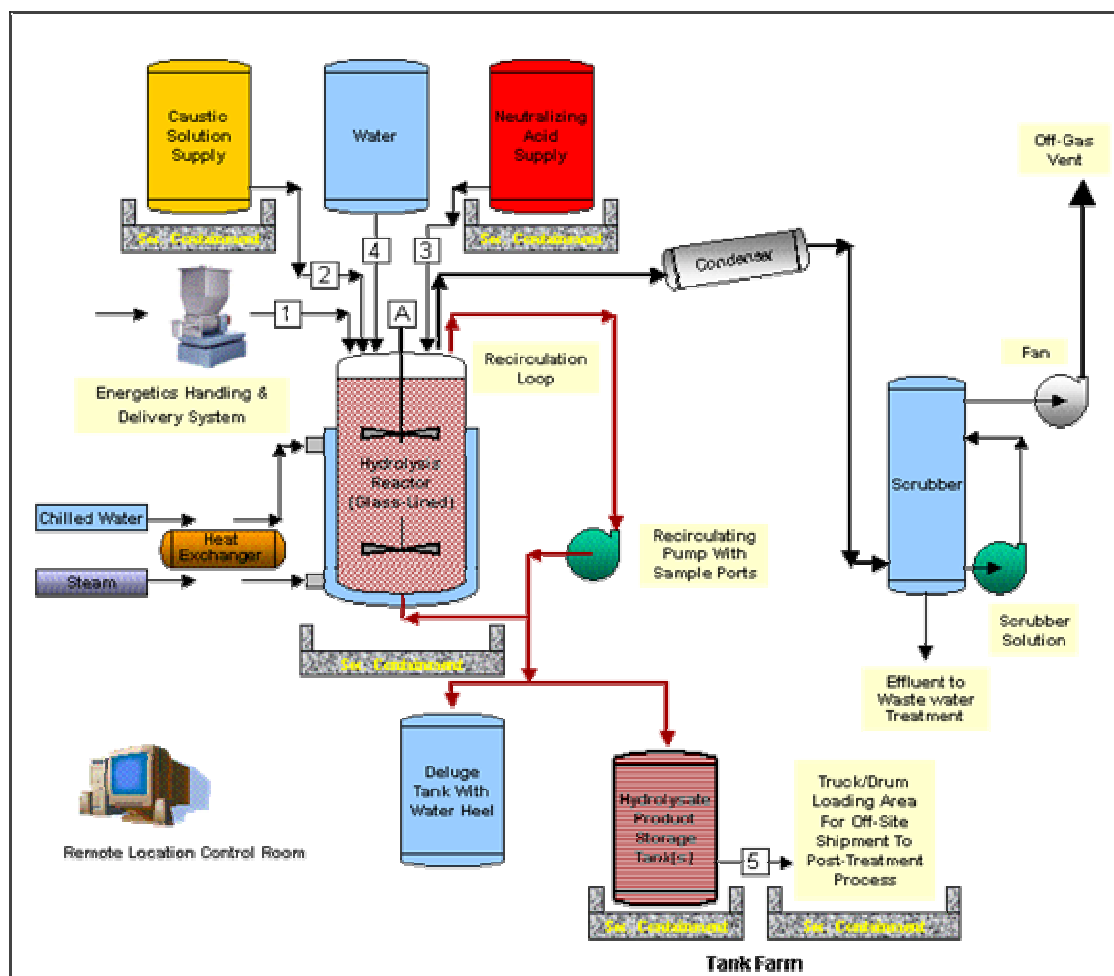


Figure 2-1. Process Schematic of the Energetic Hydrolysis Process

## **2.2 Bench Scale Testing in Response to NRC Concerns.**

NSWC and LANL were tasked to perform bench scale testing to address the NRC concerns. The thrust of this effort is:

- Evaluate temperature-time-pressure relationships of the energetic materials in aqueous alkaline solutions of sodium hydroxide
- Quantify the heat of reactions
- Determine the solubility of energetics in specific alkaline solutions,
- Assess the simultaneous processing of different types of energetics, and
- Determine the particle size reduction of energetics that must be achieved for proper post-treatment operation

LANL has issued two reports (included as Appendix C and D) detailing the bench-scale efforts:

- Bishop, R., Sanchez, J., "Alkaline Hydrolysis of Composition B, Tetrytol, M1, M8, and M28 Propellant." Los Alamos Unclassified Report, LA-UR-01-4424, Los Alamos National Laboratory, Los Alamos, NM, (2001).
- Bishop, R., Sanchez, J., "Heat of Reaction for the Base Hydrolysis of Composition B, Tetrytol, M1, M8, and M28 Propellant." Los Alamos Unclassified Report, LA-UR-01-4425, Los Alamos National Laboratory, Los Alamos, NM, (2001).

The work performed by NSWC is not being reported. The results of the accelerated colorimeter work were inconsistent, most likely due to the reactivity of the sample before insertion into the ARC.

## **2.3 M28 Surrogate Propellant and Hydrolysate.**

M28 surrogate propellant was manufactured at Radford AAP to support the technology demonstrations, as well as the testing of the hydrolysis at Holston AAP. A leaded (lead stearate per the formulation requirements) and unleaded version of the propellant was produced by Alliant Techsystems. In two cases, the propellant was hydrolyzed at Radford AAP and shipped to the technology providers (the hydrolysis system was not available at the time of these efforts) using a simple stirred heated open tank. During the second hydrolysis run, an incident occurred during the hydrolysis reaction that resulting in an over-pressure and rupturing of the piping loop supporting the hydrolysis tank. The damage to the equipment was minor. The description of the M28 manufacturing process and the incident report are included as Appendices E and F.

### 3.0 SYSTEM AND HARDWARE DESCRIPTION / OPERATIONS

The energetics hydrolysis system is comprised of the following major subsystems.

#### 3.1 Energetic Feed System.

An Acrison, Inc. Model 402-1015Z weight-loss feeder was used to feed dry energetics to the hydrolysis reactor. The unit had a 500-pound working capacity with a feed rate range of 20- to 3000-pounds per hour. The unit contained a conditioning auger in the feed bin to prevent material compaction or bridging. All parts that contacted energetics were fabricated of 304 series stainless steel. The unit was fully gasketed for water wash-down. The weigh-feeder was located on the 3<sup>rd</sup> floor in Building G-10, mounted onto a work platform. The energetics was manually charged into the hopper before the start of the test run. The energetics discharged from the metering auger fell through a 6-inch diameter stainless steel chute into the reactor (gravity feed).



Figure 3-1. Acrison Loss-in-Weight Feeder

The unit was capable of continuous or batch weighing. Located on the platform above the feed hopper was a 1-inch by 1-inch screen (stainless steel) that served as a final screening of the material before entering the bin.

The weight-feeder was isolated from the reactor using upper and lower slide-gate valves (Figure 3-2). The slide valves operated in tandem, sequenced to ensure that energetic material was not captured within the down-comer chute. All energetics materials with the exception of the M8 sheet propellant were processed using this system configuration.



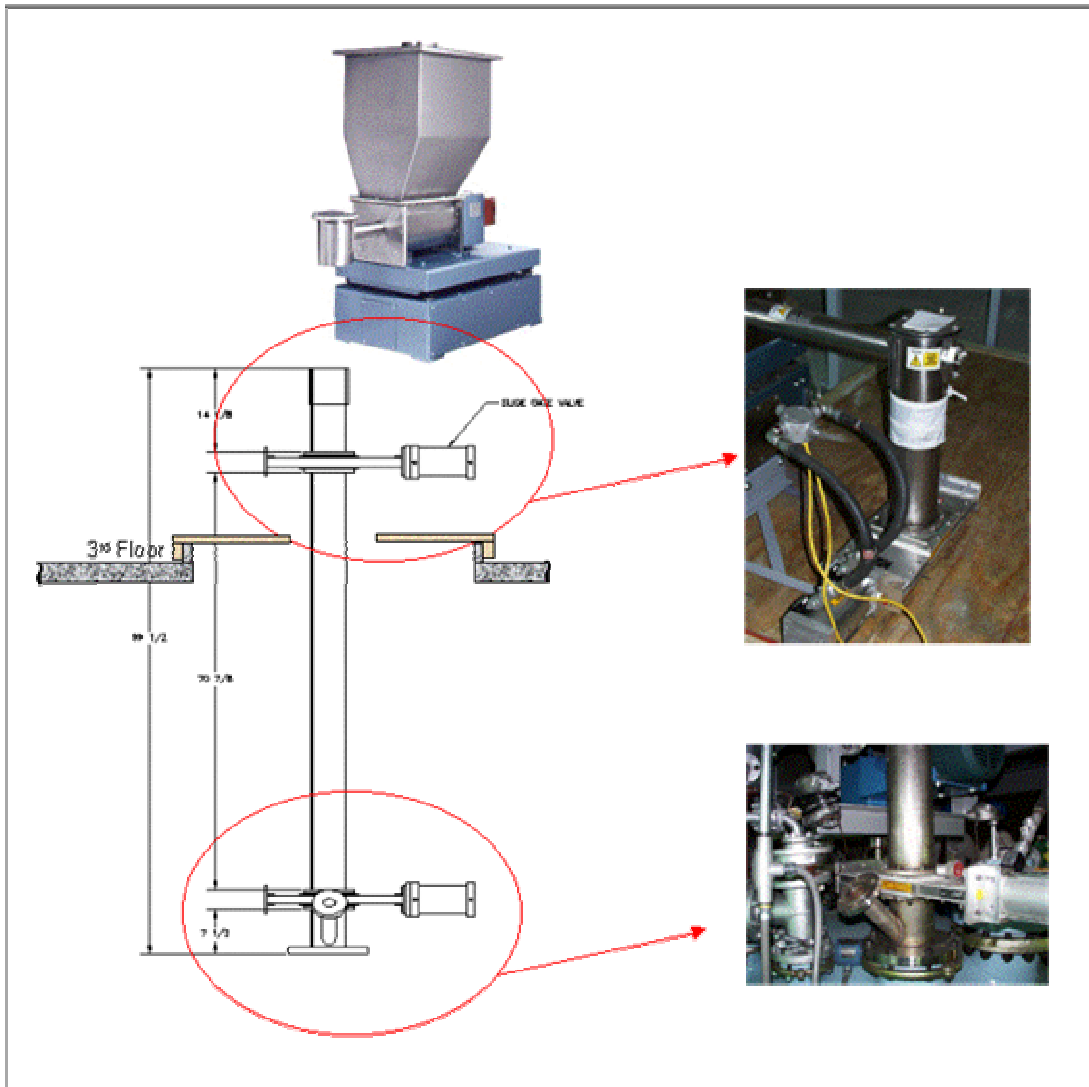


Figure 3-2. Slide Valve and Feed Chute Assemblies

The M8 sheet propellant was manually fed to the reactor, by-passing the weigh-feeder, because of its configuration. The interface between the weigh-feeder discharge and the down-comer chute was modified as shown in Figure 3-3. An operator manually fed the M8 sheet propellant into the hopper at a rate approximating the required pounds per hour feed rate selected for the test.

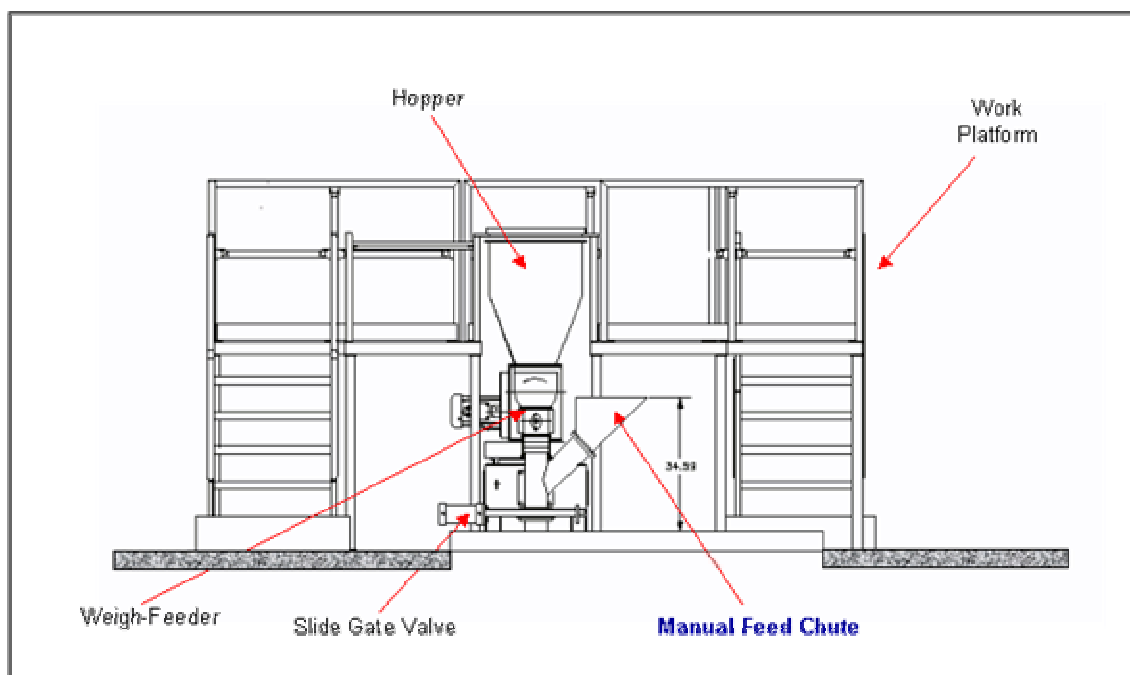


Figure 3-3. Manual Feed Chute Used with M8 Sheet Propellant

### 3.2 Tank Farm.

Sodium hydroxide and sulfuric acid and/or nitric acid are stored in the tank farm adjacent to Building G-10. The tank farm also served as a storage area for the hydrolysates produced during testing.



Figure 3-4. Tank Farm Servicing Building G-10

### 3.3 Hydrolysis Reactor.

Pfaudler, Inc. fabricated the reactor vessel complete with structural supports, work platform and walkway. The vessel is a Pfaudler RS-78-2000-125-100 glass lined carbon steel RS-Series reactor; 2000-gallon capacity; 78-inch diameter by 84-inch straight side; conventional single chamber carbon steel jacket; ASME design and stamp for 125 psig/FV at -20 to 450°F internal and 100 psig/FV at -20 to 450°F jacket (90 psig with full internal vacuum); with 9115 blue Glasteel® surface. The cover contains two, 10-inch flanged ports; one 8-inch flanged ports; one, 6-inch flanged ports; and five, 4-inch flanged ports. The heating capability of the reactor is 1,500,000 BTU/hr; the cooling capability is 500,000 BTU/hr.



Figure 3-5. Pfaudler Hydrolysis Reactor (Typical Representation)

The system as delivered partially assembled from Pfaudler, Inc. and included following design features and ancillary systems:

- Interior surfaces configured to minimize accumulation of precipitates
- Glasteel® cover-mounted baffles to promote mixing/distribution of energetics
- Fin-type Glasteel® baffle with tantalum encased RTD
- Tachometer for monitoring and controlling agitator speed
- Great Lakes Model 692P Two-Wire pH Transmitter
- Krohne Level-Radar Sensor BM 70 A
- Chemineer Model 2HTD-10 Turbine Agitator, Hastelloy C-276, Turbofoil upper and lower pitched-blade impellers (10 HP)
- Toshiba variable frequency controller (VFC) for use with Chemineer Agitator System
- Rosemount Series 8700 Magnetic Flowmeter Systems for caustic, acid and water
- Heat Exchanger – Reactor Jacket, Kam Thermal Equipment Ltd. (143 ft<sup>2</sup>), 304L stainless steel tube side, carbon steel shell
- Heat Exchanger – Condenser (43 ft<sup>2</sup>) with Hastelloy C-276 tube side, carbon steel shell
- Dual discharge port valves
- Process piping 316L stainless steel, Hastelloy C-22, Teflon lined
- Feed ports and analytic sampling ports for solids and liquids
- HYL80 Toroidal Explosion-proof Process Light
- Manway cover, 24-inch diameter (spring assist with fused sight glass) for maintenance
- Auxiliary water seal assembly per RONA design requirements
- Reactor support frame and work platform with walkway

To avoid building pressure within the reactor from off-gassing released during the base hydrolysis reaction, outside air was continuously be drawn through the vessel carrying the off-gassing from the reactor to a condenser and then on to a scrubber/stripper/absorber system before venting to atmosphere to ensure that no toxic chemicals are released to atmosphere. The off gassing was analyzed for the presence of NO<sub>x</sub>, CO<sub>2</sub>, CO, TOC, and others using online analyzers (see paragraph 3.9 below).

The design of the agitator was determined by Pfaudler, Inc. based on the volume of the reactor vessel and the requirement to ensure that the solution is maintained homogenous throughout hydrolysis reaction. The detailed specifications and drawings for the Pfaudler reactor including ancillary equipment are provided in Appendix G.



Figure 3-6. Turbofoil Pitched-Blade Agitator

A pH control system was installed on the reactor to maintain a required/specified pH for post-treatment operations. The unit was located in the recycle line. However, as expected, at the higher caustic levels, pH greater than 9, the sensor would go off-scale and proved useless with regard to being used as a process control and monitoring device. Therefore, the installation of the pH meter was more experimental in nature (to assess hardware performance as a potential means of controlling the pH of the hydrolysate solution and the neutralization process) as opposed to being needed for process control.

### 3.4 Liquid Sampling System.

A liquid sampling system, Intersystems Sampling System, Model LF, was flange-mounted on the recirculation line. The sampling probe is 1-inch in diameter fabricated of 316 series stainless steel with TFE seals. Each sample is approximately 10-ml. The sampling rate is programmable from 0.01 to 999 hours. The unit is supported by a 16-station carousel mounted in an enclosure capable of being chilled using ice or dry ice. All controls are explosion-proof. The pressure rating of the sampler is 150 psig and the temperature rating is 500°F.



Figure 3-7. Liquid Sampling System

There are sixteen index positions on the carousel; each position has a 250-ml HDPE sample bottle into which the sample is drained.

#### 3.4.1 Typical Sampling Procedure:

The following procedures was developed based discussions with A.D. Little, Inc. and LANL:

Sample Bottle Preparation: In an attempt to maintain the hydrolysate sample at the conditions at which it was taken, the collected samples were quickly quenched in sulfuric acid and chilled to ~4°C in ice. To accomplish this, an acid heel is placed in each sample bottle before the bottle is mounted on the carousel, typically 30 ml of sulfuric acid (6 normal). The cabinet is packed with ice to maintain the ~4°C temperature.

Sampling: A single sample was comprised of SIX sample aliquots that were injected into the sample bottle on the carousel (the sampler was programmed to cycle the injector six times). The total volume of these six sample aliquots would be approximately 42 milliliters (i.e., ~7 ml per sample aliquot).

Flush: After the six aliquots were injected into the sample bottle, the sampler / tubing was flushed with 100 ml of distilled / deionized water to "clean" the system into the sample bottle. The water was fed into the sample line immediately below the sampling valve to ensure that the water would "flush" across all of the areas that had been "wetted" by the hydrolysate sample. After the flush, the sample carousel would be indexed to the next sample position and the sampling sequence repeated.

The total volume in the sample bottle as it was taken from the sampler unit was about 172 ml consisting of 100 ml flush water, 30 ml of 6N sulfuric acid heel, and approximately 42 ml hydrolysate sampled from the reactor. Typically, sixteen samples would be collected during an experimental run. The samples are packaged per A. D. Little, Inc. specification and sent to an independent laboratory through A. D. Little, Inc. for analysis and reporting of the results, as required by the test protocols establish for the ACWA program.

#### **3.4.2 Hardware Modifications:**

Two modifications were made to the liquid sampling system to improve performance: A purge line was installed into the system immediately below the sampling valve in the recirculation line so that the sampling mechanism and line could be flush with distilled / deionized water to clean the system and prevent / minimize "carry-over" between samples. A vessel of distilled / deionized water (20 liter Nalgene bottle) was stored on the third floor of Building G-10. By remotely opening a needle valve for a specified time period (typically 15 seconds, which correlated to about 100 ml of water), the water would be gravity fed into the system as the purge. A small hole (1/16-inch) was drilled in all of the plastic holders / lids (16 in total on the carousel) into which the 250-ml sample bottles were threaded and suspended. This hole provided venting for the bottles during the introduction of liquid hydrolysate or flush, which prevented the sample bottles from pressurizing.

#### **3.5 End of Run Liquid Sample.**

At the end of each experimental run, a bulk sample would be taken using a series of valves, which could be opened in the recirculation line (while the hydrolysate was being pumped through the line). In practice, approximately 4 liters of the hydrolysate would be collected in a 4-liter volume HDPE jug to "flush" the line / valve and then discarded. Immediately after this flush, hydrolysate would be collected in additional jug(s) as the "end-of-run" sample. The volume of this "end-of-run" sample would be either 4 liters or 8 liters as specified by A. D. Little, Inc. personnel.

### 3.6 Control System.

The energetics hydrolysis process in Bldg. G-10 was fully automated and remotely controlled from the Central Control Building, Building 155 via a fiber optic link to the process area. The process displays were generated using PCS7-WinCC software.

Figure 3-7 shows the operator in the Control Room interfaced to the process floor along with process flow diagram displays of the controlled and operating process parameters



Figure 3-8. Central Control Room, Process Displays

The PCS7-WinCC software was used to plot key process operating and control parameters for each run of the runs reviewed in paragraph 6 (unless otherwise noted). An example of this chart is shown in Figure 3-8 (the operating data for commissioning Run 4, Composition B explosive) where:

- **Energetic Feed Rate:** This will normally be represented as a step function over a four-hour period corresponding to the feed rates cited above.
- **Hydrolysate Temperature:** This represents the temperature of the hydrolysate in the reactor during the process.
- **Reactor Jacket Temperature:** This is the temperature of the cooling/heating medium within the jacket of the reactor.
- **Reactor Outlet Flow:** This is the airflow through the reactor headspace to the scrubber. The airflow was maintained at ~40 scfm throughout the tests
- **Reactor Air Sweep:** This is the amount of air flowing into the reactor headspace during the processing of the energetics.

The data logger ran continuous both while the process was underway and during non-processing periods. In addition to the data presented on the charts, the agitator speed, valves settings,



energy consumption of pumps, level flow rates, etc. were logged continuously and could be plotted “real time” at the discretion of the operator to evaluate trends and/or create a hard copy of specific test data..

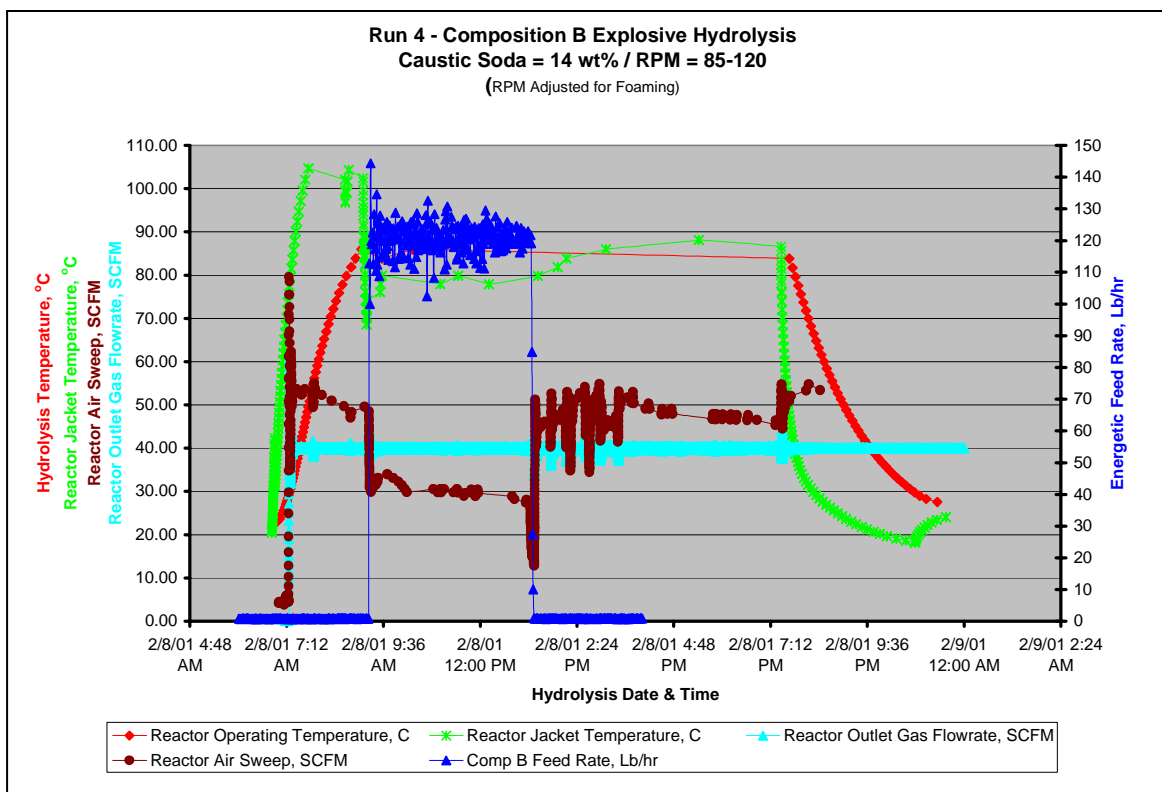


Figure 3-9. Process Operating and Control Parameters

The plot presents the explosive addition at a rate of ~125 lbs/hr over a four hour period, the hydrolysate temperature versus the jacket temperature, and the sweep air and make-up air flowing across the reactor head space. The agitator speed was varied during the run between 85-to-120 rpm to control the foaming that was encountered during process commissioning. Data can also be recovered for the rpm, current draw on the recirculation pump, etc. These processing parameters may be plotted against time. However, to keep the chart simple, only the five process variables presented above were plotted for each of the test runs.

### 3.7 Process Description.

The alkaline solution will be introduced into the reactor from the tank farm adjacent to Building G-10, the pH adjusted through introducing process water to the caustic solution, and heated to a desired temperature by circulation steam or heating fluid through the reactor jacket. While the caustic solution is being prepared, the energetic material to be process will be charged into the weigh-feeder hopper located on the 3<sup>rd</sup> floor. When the caustic solution reaches the required temperature, the energetic materials are added into the reactor using the weigh-feeder system on the 3<sup>rd</sup> floor.

During the addition of the energetic material, a misting spray is used inside the reactor to control any dusting that may occur as the energetic material falls through the feed chute into the reactor. Throughout the addition period and the hydrolysis reaction, the caustic solution is vigorously agitated while maintaining the temperature at a desired set point for several hours during which time the energetic materials are completely hydrolyzed. Vigorous agitation is required to ensure that all energetic particles are exposed to complete hydrolysis.



During the hydrolysis reaction, air and liquid samples are taken to monitor the progress of the reaction and quantify the off gassing that occurs. The following process parameters are monitored and recorded continuously throughout the operation:

- Dump Tank Temperature
- Dump Tank Level
- Reactor Head Space Temperature
- Caustic Storage Tank Level
- Acid Storage Tank Level
- Circulation Flow
- Circulation Temp
- Reactor Temperature, Primary
- Reactor Temperature, Redundant
- Circulation Loop Temperature
- Reactor pH
- Building 15-lb. Steam Temperature
- Reactor Water Spray Flow
- Reactor Acid Spray Flow
- Reactor Cooling Water Temperature
- Reactor Agitator Speed AG-1
- Scrubber Fan Speed
- Dump Tank Agitator Speed
- Loss-in-Weight Feeder Speed
- Reactor Overflow Line Pressure
- Steam Condenser Level
- Reactor Level
- Caustic Flow
- Acid Flow
- Water Flow
- Air Flow Into Reactor
- Air Flow To Scrubber
- Circulation Pump Amps

At the conclusion of the hydrolysis reaction, the hydrolysate is allowed to cool to ~35°C. The hydrolysate will be held in the reactor, with continuous agitation, to conduct chemical analysis to characterize the product before releasing to a holding/storage tank to be processed in post-treatment process. If the hydrolysate solution is too alkaline, an acid will be added to control pH to the specified post-treatment process.

The standard operating procedure (SOP) that was developed and validated for the energetics hydrolysis system is including as Appendix H.

### **3.8 Failure Mode and Effects Analysis (FMEA).**

At the conclusion of the design phase of the program, an FMEA was performed to identify any operational and/or safety issues so that corrective actions could be taken before the reactor system and ancillary hardware was delivered to Holston AAP. Upon receipt and installation of the reactor process system, a second FMEA was performed on the as built/as installed system including all infrastructure support the operation of the hydrolysis system. The results of the two FMEAs are provided in Appendices I and J.

### 3.9 Analysis of Off Gassing.

The off-gas from the reactor was continuously analyzed to determine its composition (see Figure 3-9 for the schematic of the off-gas analysis system and Appendix K for a description of the analyses performed by the system).

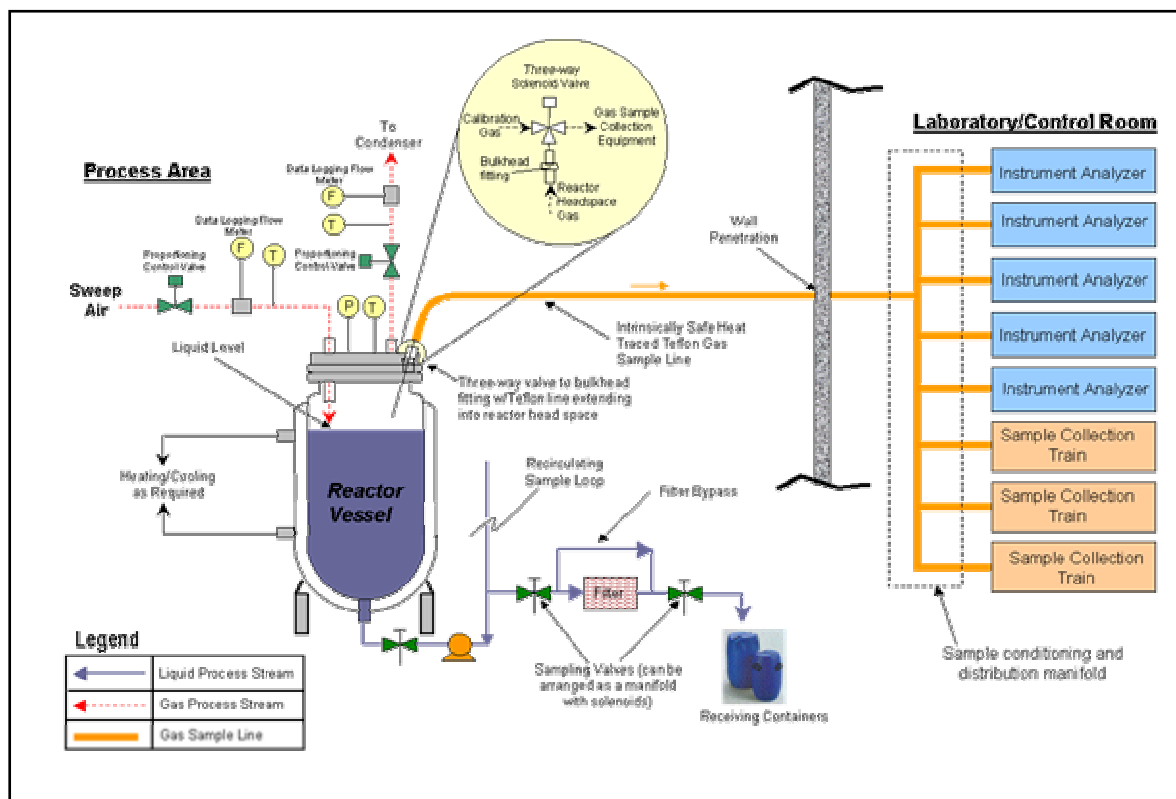


Figure 3-10. Schematic of Off-Gassing Monitoring System (Real Time)

A 1-inch inside diameter Teflon® sample probe is positioned in the gas stream at the exhaust vent of the Reactor Vessel immediately exit of the headspace of the vessel. This probe is connected to three separate Teflon® sample lines approximately 350 feet in length. The lines are steam traced inside the building (about 150 feet) and electrically traced once they exit the restricted area of the building (about 200 feet) and are maintained at approximately 225-250 °F to prevent condensation of moisture (or organic compounds) during transport. The three lines have the following function:

- Line 1: The Batch Train Sample line – ½-inch ID Teflon sample line used to transport approximately 20 Liters/min of headspace gas to the individual batch trains.
- Line 2: The CEMS Sample line – 3/8-inch ID Teflon sample line used to transport approximately 10-15 liters/min of headspace gas to the CEMS analyzers.
- Line 3: The CEMS Calibration line – 3/8-inch ID Teflon sample line used to transport calibration gas from the mobile laboratory to the sample valve and back to the CEMS analyzers in order to calibrate the analytical instruments.

### 3.10 Engineering Design Package.

The engineering design package for the as-installed 2000-gallon hydrolysis reactor and ancillary supporting systems is provided in Appendix L

#### 4.0 MATHCAD™ MODEL & SIMULATION

Of particular interest to the design engineers are the heats of reaction of the individual energetic materials being hydrolyzed. A MathCAD™ model was developed by LANL to estimate the heat released during the hydrolysis of each energetic material and compared to actual lab measurements. The following is extracted from the LANL report:

The heat of reaction was measured using a simple, small-scale, differential-thermal-analysis method. Heat of reaction averaged over the entire run, and heat of reaction at peak reaction temperature. The second is a higher number and should probably be used for safety and design calculations. For reference, similar studies gave the heat of reaction for HMX at 1.5 kJ/g. Other methods for HMX give values of 2.1 and 2.3 kJ/g. Therefore, this method does not give the most conservative answer. The design number should be estimated as 25-35% higher to account for energy loss due to vaporization and/or boiling.

The results using this method had a large amount of variation between samples, and in some cases were difficult to interpret due to foaming and/or boiling problems. However, this data was found useful for a first approximation of the heat liberated during the base hydrolysis reaction. The heat of reaction information, along with previous reaction rate and product information was integrated into a Mathcad™ program to predict products and heat produced during a large-scale hydrolysis run. The information should be used to aid in the scale-up and design of future reactors.

Finally, base hydrolysis data obtained from these two studies was used to determine the thermal runaway temperature threshold for all five explosive and propellants studied. The thermal runaway calculations show that there should be no safety problems if the hydrolysis reactions are run below 130°C. This is well above any temperatures postulated for any atmospheric reactor design.

Table 4-1. Summary of Heats of Reaction

Explosive	NaOH Concentration	$\Delta H_{rxn}$ kJ/g (average) $\pm$ standard deviation	$\Delta H_{rxn}$ kJ/g (peak) $\pm$ standard deviation
M1	12 wt%	0.151 $\pm$ 0.009	0.34 $\pm$ 0.038
M1	20 wt%	0.23 $\pm$ 0.011	0.59 $\pm$ 0.018
M1	35 wt%	0.237 $\pm$ 0.006	1.3 $\pm$ 0.13
M8	12 wt%	Boiled Over for All Flasks	0.94 $\pm$ 0.18
M8	20 wt%	0.228 $\pm$ 0.02	0.89 $\pm$ 0.09
M8	35 wt%	0.211 $\pm$ 0.18	1.39 $\pm$ 0.12
M28	12 wt%	0.115 $\pm$ 0.005	0.35 $\pm$ 0.028
M28	20 wt%	0.12 $\pm$ 0.08	0.38 $\pm$ 0.14
M28	35 wt%	0.38 $\pm$ 0.08	1.06 $\pm$ 0.36
Comp B-4	12 wt%	0.211 $\pm$ 0.006	0.53 $\pm$ 0.041
Comp B-4	20 wt%	0.187 $\pm$ 0.004	0.67 $\pm$ 0.07
Comp B-4	35 wt%	0.34 $\pm$ 0.06	0.90 $\pm$ 0.05
Tetrytol	12 wt%	0.23 $\pm$ 0.05	0.75 $\pm$ 0.07
Tetrytol	20 wt%	0.25 $\pm$ 0.09	0.83 $\pm$ 0.12
Tetrytol	35 wt%	All Flasks Foamed Over	0.81 $\pm$ 0.13

The Mathcad™ simulation model can be used to support scale-up for design purposes so long as the geometry of the reactor remains the same.

## 5.0 PILOT PLANT COMMISSIONING

The energetics hydrolysis system was commissioned using Composition B explosive (nominal composition is 60% RDX {includes HMX percentages varying from 5-20%}, 40% TNT, and plus 1% wax added) to gain operational experience on all unit operations and to verify that the controls and instrumentation was working properly. The working level in the reactor was ~1700-gallons for all commissioning runs, which represented the vendor recommended 80% of reactor volume.

### 5.1 Commissioning Run 1.

Commissioning runs with 200-pounds of Composition B explosive commenced on 14 December 00. Problems were encountered with the Acrison weigh-feeder, which shutdown almost immediately upon starting the feed cycle. Numerous restarts were required before the weigh-feeder would operate. For safety reasons, operators must enter the process area to reset the control panel when the weigh-feeder shuts down – the reset cannot be performed remotely. This problem would recur throughout the commissioning runs, as well as during the test program until the problem could be effectively trouble shoot and corrected.

#### 5.1.1 Weigh-Feeder System:

The shutdown was caused by excessive current draw during the start-up of the drive motor that turns the conditioning and feed augers. The current draw was verified to be correct, peaking at nearly 95% of the maximum. Initially, weight-loading of the energetics on the conditioning and feed augers was thought to be the problem since reducing the amount of explosive in the bin to less than 150 pounds allowed the system to run without the overload at start-up. However, the system was design to handle this amount of weight (and greater weights), and the test program could not be executed efficiently with setting a 150-pound limit on the weigh-feeder. The settings were adjusted to minimize load sensitivity and a high start-up feed rate was used on the recommendation of the vendor. This did not solve the problem but minimized the occurrences, allowing the testing to proceed with minimal schedule delays.

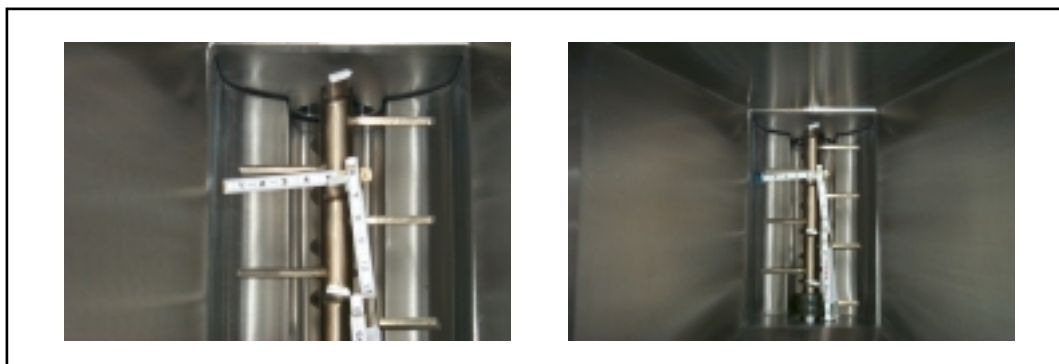


Figure 5-1. Weigh-feeder Modified Conditioning Auger

Finally, through trial and error as the test program proceeded, the conditioning auger design was identified as the cause of the problem. The conditioning auger is used to prevent bridging of the material in the bin. Because energetics are considered non-powdered materials, the vendor selected a blade design. However, the clearances between the blade tips of the conditioning auger and the side-mounted bin tabs were only 0.125-inches. Under certain situations, the energetics would settle in the bin and become trapped between the auger blade and sidewall tab causing an immediate current draw at start-up. (Note: This was not an explosion hazard since the force on the energetics never materialized.) The auger blade was not angled, which further aggravated the problem since the flat blade had to push through the material in the bin, placing a large load on the motor at start-up. The blade should have been slightly angled so as to pass more easily through the material and thereby reducing the load on the motor. The conditioning auger was removed and the auger tips shortened by 0.750-inches increasing the clearance from 0.125 to 0.875-inches. This modification appeared to solve the problem.

### 5.1.2 Test Results:

The plots present Composition B explosive destruction as a function of reactor residence time. Hydrolysate samples were taken during both the addition and the reaction periods. The peaks on the plots show high energetics concentration during the first four hours addition time.

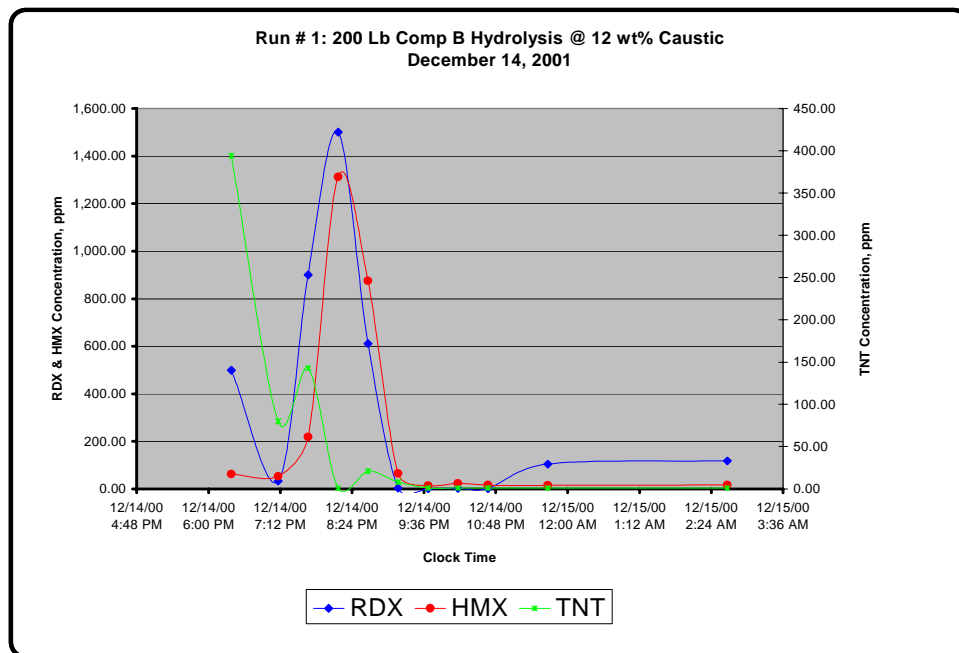


Figure 5-2. Run 1, Mid-Run Destruction Efficiency

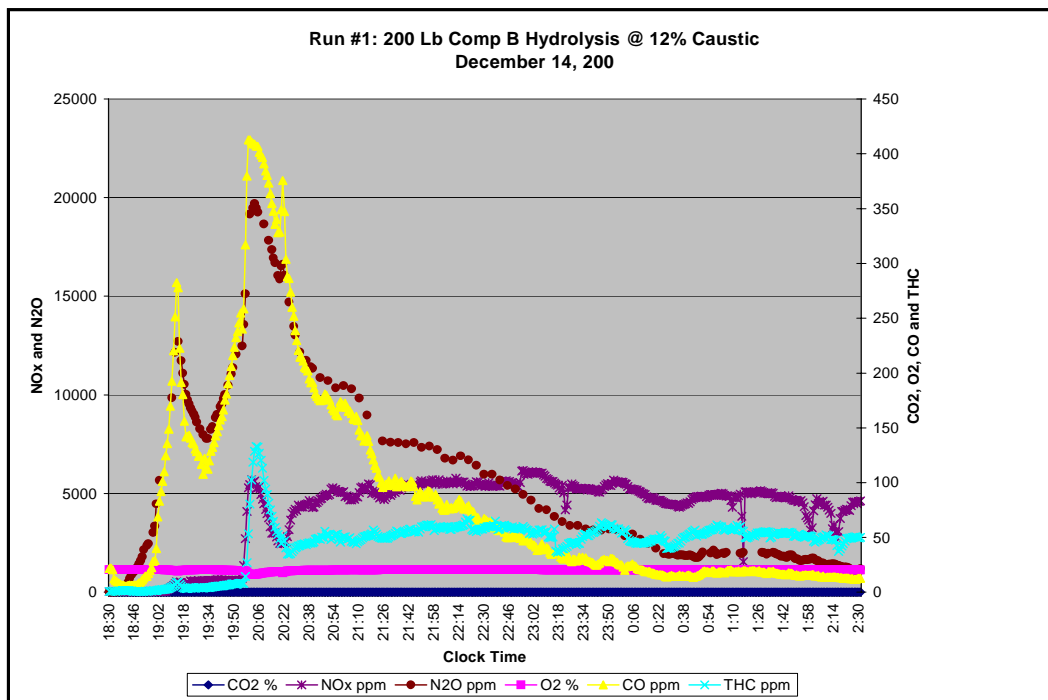


Figure 5-3. Run 1, Off-gas Production

Table 5-1. Run 1, Composition B Off-gas Analysis

Reactor Off Gas Analysis					
Component	During Energetic Addition	Note	During Reaction	Note	Unit
1,2-Dichloroehtene (total)	5.600		4.800	U	ppbv
1,3,5-Trinitrobenzene	6.130	MAX			ug/m <sup>3</sup>
2,4,6-Trinitrotoluene	617.000				ug/m <sup>3</sup>
2,4-Dinitrotoluene	45.600				ug/m <sup>3</sup>
2,6-Dinitrotoluene	13.700	MAX			ug/m <sup>3</sup>
2-Butanone	58.400	J	340.000	J	ppbv
4-Amino-2,6-Dinitrotoluene	8.650	MAX			ug/m <sup>3</sup>
Acetaldehyde	599.000		699.000		ug/m <sup>3</sup>
Acetone	828.000		1,820.000		ppbv
Ammonia	372,000.000		3,540,000.000		ug/m <sup>3</sup>
Benzene	38.300		34.900		ppbv
Butanal	81.000		49.200		ug/m <sup>3</sup>
Carbon Dioxide	0.080		0.110		%
Carbon Monoxide	103.000		83.400		ppmv
Chloroform	5.600	U	10.600		ppbv
Crotonaldehyde	2.840	U	59.700		ug/m <sup>3</sup>
Cyanide	0.011	U	0.003	U	ug/m <sup>3</sup>
Cyclohexanone	2,650.000		224.000		ug/m <sup>3</sup>
Decanal	252.000		126.000		ug/m <sup>3</sup>
Dibromochloromethane	12.100	J	4.800	UJ	ppbv
Ethylbenzene	5.600	U	11.600		ppbv
Formaldehyde	1,960.000		7,730.000		ug/m <sup>3</sup>
Hexanal	7.370	J	12.100		ug/m <sup>3</sup>
Methylene Chloride	25.600	J	56.600		ppbv
m-Tolualdehyde	20.300	J	1.730	U	ug/m <sup>3</sup>
Nitrous Oxide	6,934.000		6,132.000		ppmv
Nonanal	92.700		3.030	U	ug/m <sup>3</sup>
NOx	275.000		5,018.000		ppmv
Octanal	3.360	U	50.800		ug/m <sup>3</sup>
Pentanal	1.700	U	50.300		ug/m <sup>3</sup>
Propanal	3.890	U	42.400		ug/m <sup>3</sup>
RDX	22.700	MAX			ug/m <sup>3</sup>
Tetrachloroehtene	9.300	J	7.900	J	ppbv
Toluene	13.900	J	1,060.000	J	ppbv
Total Hydrocarbons	4.400		81.200		ppmv
Xylenes	10.900		50.600		ppbv

J = Estimated Value; concentration is below limit of quantification

MAX = Reported result was from a multi-fraction gas sampling train that contains both non-detected results and positive results

U = Analyte was not detected

### 5.1.3 Hydrolysate Neutralization:

The plan was to neutralize each batch of hydrolysate before pumping the hydrolysate to the tank farm where it would be stored until disposed of as waste. At the conclusion of Run 1, the neutralization of the hydrolysate was initiated the following day using concentrated sulfuric acid (98+%) introduced through the water spray nozzle at up to 20-gallons per minute. Approximately 10% excess NaOH was in the hydrolysate at the end of the hydrolysis reaction.

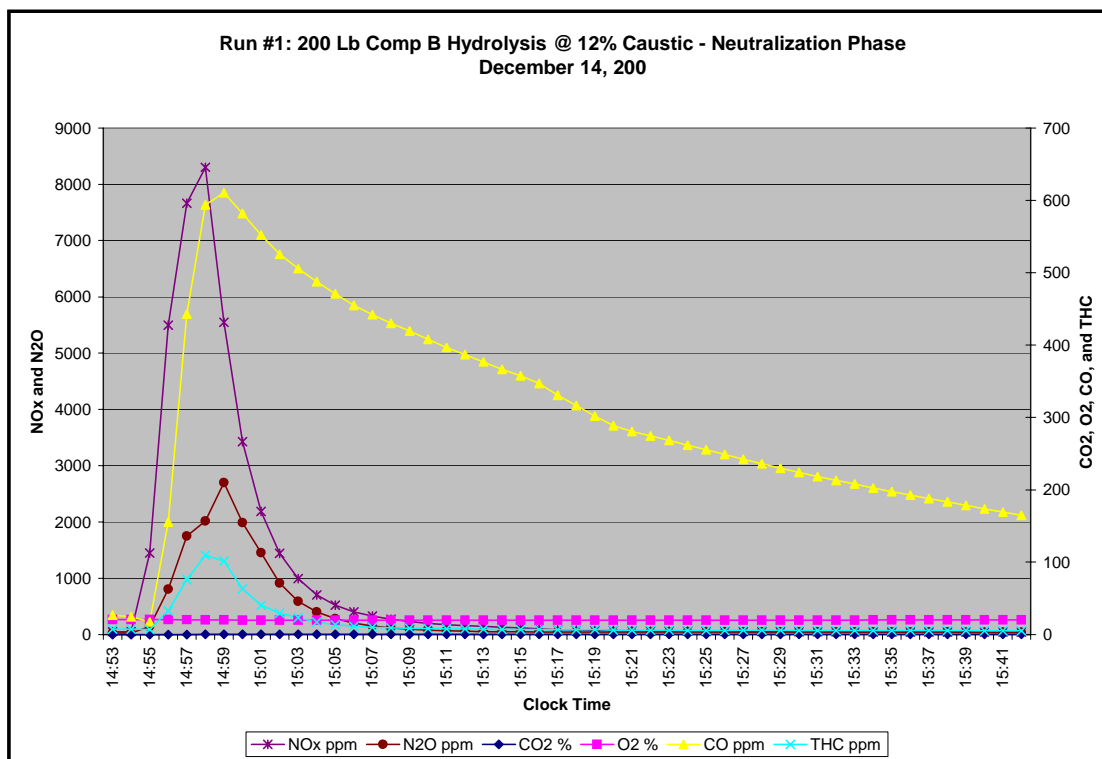


Figure 5-4. Run 1, Composition B Hydrolysate Neutralization Phase

The neutralization reaction is extremely exothermic resulting in an  $\sim 4^{\circ}\text{C}$  temperature rise in  $\sim 4$  minutes, liberating a large amount of NOx and  $\text{N}_2\text{O}$  that was seen exiting the scrubber exhaust (see Figure 5-4 above). Also, it is possible that even the more stable nitrates / nitrites in the hydrolysate were decomposed at the interface where the acid is being added (rapid pH change combined with highly localized heating, probably flash-boiling; with  $\gg 100^{\circ}\text{C}$  for a split-split-second).

The neutralization effort was aborted when the acid feed pumped failed (the failure was unrelated to the neutralization process). To avoid schedule delays, it was decided to store the hydrolysates until the testing was completed and equipment improvements could be implemented to the acid feed system to better distribute the acid, and to the scrubber system to handle the NOx emissions. The neutralization would be conducted in bulk at the end of the test and evaluation program. This decision ultimately proved to be a fortuitous since the hydrolysates produced during the test and evaluation program would now be required to support the SCWO testing at DPG.

Neutralization would not normally be performed in the chemical agent disposal facilities since the energetic hydrolysate would proceed to the final treatment step. Furthermore, maintaining the hydrolysate at a pH greater than 9 is important. The hydrolysates, if neutralized to a pH of 4 or lower, will off-gas quite vigorously. This is attributed to the large amounts of sodium nitrite and nitrate in the solution that can be decomposed in the presence of strong mineral acids, such as sulfuric or nitric acids.

#### 5.1.4 Temperature Control – Reactor Cooling Jacket:

While the cooling capacity of the heat exchanger system was more than adequate to handle the exothermic hydrolysis reaction, the temperature control system for the jacket heat exchanger did not perform satisfactory. The control loop cycled between extremes of demanding full cooling to demanding full heating resulting in over-shooting and under-shooting the set point (87°C). This came as a surprise since controlling to a set point should not have presented any challenges to the software. Evidently, the control philosophy was flawed in the sense that there were no dampening features as the actual temperature approach the set-point temperature. As a result, the temperature of the jacket was controlled manually through the control panel.

#### 5.1.5 Mass Flow Meter:

The mass flow meter was installed to monitor changes in the fluid properties of the hydrolysate as the hydrolysis reaction proceeded to conclusion. The meter was installed on the suction side of the recycle pump. The meter performed acceptable during equipment debug and set-up (water and caustic solution). Shortly after the addition of the Composition B explosive was initiated, the meter reading went off-scale, initially leading the operators to believe that the recycle line had become clogged. However, the current draw on the recycle pump was normal, and there was no temperature change in the loop. After the addition of the Composition B was completed, and the hydrolysis reaction had been under way for 6 hours, the readings on the mass flow meter returned.

The cause of the meter malfunction was aeration of the suction side of the recirculation loop and the off-gassing taking place from the hydrolysate solution during the digesting of the explosive. The mass flow meter should have been installed on the discharge side of the line, and a de-aerating device installed in the line to protect the meter. This failure occurred again on Runs 2 and 3. The aggressive schedule did not allow time to correct the problem before testing was concluded for the program. While the failure of the mass flow meter did not impact the processing studies, the opportunity to obtain “nice to have” information regarding the characterization of changes in fluid viscosity as the reaction proceed with different caustic strength and energetic loadings was lost.



## 5.2 Commissioning Run 2.

Commissioning Run 2 was conducted with Composition B explosive fed at a measured rate of up to 492 lbs/hr to the reactor – the maximum rate anticipated for energetics in a full-scale chemical weapon demilitarization facility. Based on experiences of Run 1, the jacket temperature was manually controlled and liquid samples would again be taken during the explosive addition. The same feeder problems occurred as were encountered on Run 1. The mass flow meter malfunctioned shortly after the addition of the explosive was initiated, confirming believe that the recycle line was aerating, and possible off gassing of energetics was taken place in the recycle line as the hydrolysate flowed through the pipe.

### 5.2.1 Test Results:

The following charts plot the destruction of the Composition B explosive. Figure 5-5 tracks the destruction of the Composition B explosive against time.

The significant rise and fall of the HMX and RDX concentrations shown is probably the result of HMX and RDX being freed from the TNT as the TNT is being melted and hydrolyzed, and then entering the recycle line as the hydrolysis proceeds to conclusion. This became the typical cyclic trace for all samples taken during the addition process.

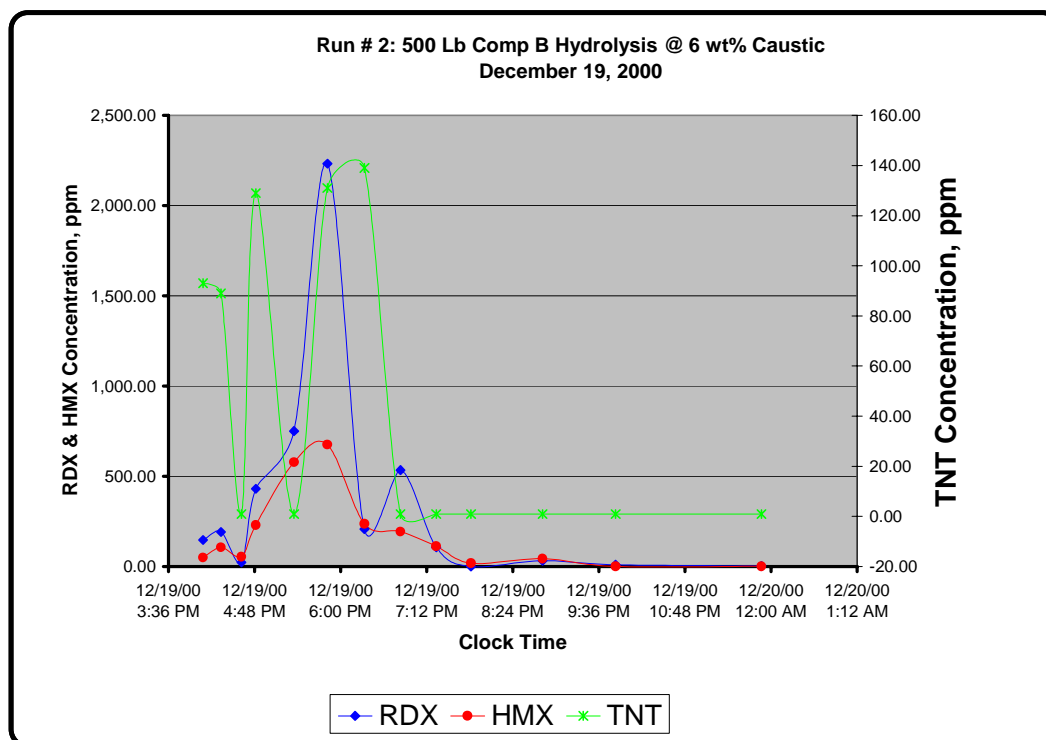


Figure 5-5. Run 2, Mid-Run Destruction Efficiency

Figure 5-6 shows the off gassing that was occurring during the hydrolysis reaction. The repeated stopping and starting of the weigh-feeder as the operators struggle to keep the feed system operating caused the multiple spikes in the traces for NO<sub>x</sub>, N<sub>2</sub>O and CO. The off gassing rate decreased when the feeder stopped, and as soon as the feeder was restarted the off gassing rate increased.

Although unintentional, this shows how closely the off gassing tracked with the rate of energetics addition and the destruction of the energetic.

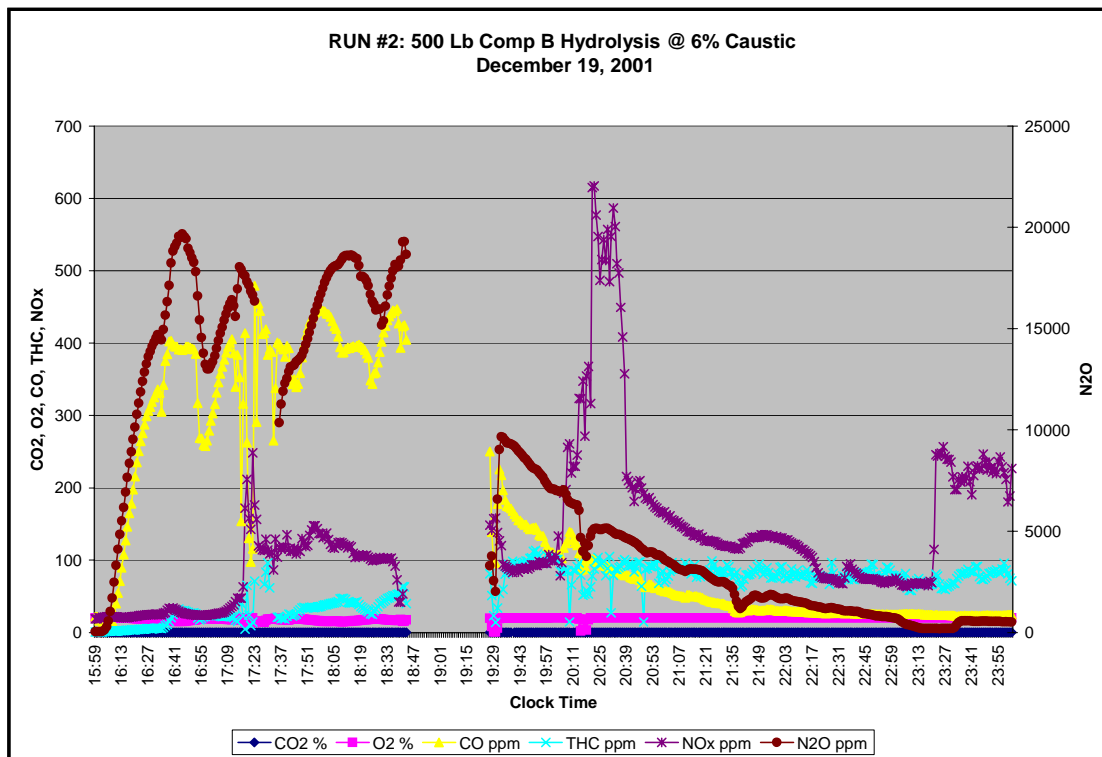


Figure 5-6. Run 2, Off-gas Production

During Run 2, severe foaming was encountered that eventually contaminated the air sampling lines. The agitation in the reactor was increased to bring the foam under control, indicated by the spiking of the NOx trace. The foaming problem was aggravated because the level within the reactor was extremely high – at least 18-inches above the upper agitator, consequently, the vortex was nearly non-existent. The additional water entering the reactor through the spray nozzle used to prevent dusting of the explosive as the explosive fell into the reactor caused the high level.

Run 2 was the last run conducted in calendar year 00. Testing was not resumed until February 01. TRC took the break in testing as an opportunity to clean the sampling lines, which were contaminated by the foaming and particle deposition. The lines were flushed using DI water and solvent followed by a nitrogen purge. The Teflon probe was increased from 0.375-inches diameter to 1.0-inch diameter.

The concentration of the major components of the off gas stream is provided in Table 5-2.

Table 5-2. Run 2, Composition B Off-gas Analysis

Reactor Off Gas Analysis					
Component	During Energetic Addition	Note	During Reaction	Note	Unit
1,3,5-Trinitrobenzene	18.20	MAX			ug/m <sup>3</sup>
2,4,6-Trinitrotoluene	3,140.00				ug/m <sup>3</sup>
2,4-Dinitrotoluene	314.00	MAX			ug/m <sup>3</sup>
2,6-Dinitrotoluene	2,730.00				ug/m <sup>3</sup>
4-Amino-2,6-Dinitrotoluene	148.00	MAX			ug/m <sup>3</sup>
Acetaldehyde	595.00	J	1,120.00	J	ug/m <sup>3</sup>
Ammonia	1,580,000.00		12,000,000.00		ug/m <sup>3</sup>
Benzene	45.70		35.40		ppbv
Butanal	0.39	UJ	2.63	UJ	ug/m <sup>3</sup>
Carbon Dioxide	0.05		0.07		%
Carbon Monoxide	313.40		55.60		ppmv
Crotonaldehyde	250.00	J	401.00	J	ug/m <sup>3</sup>
Cyanide	0.01		0.30		ug/m <sup>3</sup>
Cyclohexanone	1.21	UJ	8.29	UJ	ug/m <sup>3</sup>
Decanal	227.00	J	60.50	UJ	ug/m <sup>3</sup>
Dibromochloromethane	13.20		17.50	U	ppbv
Formaldehyde	137,000.00	J	749,000.00	J	ug/m <sup>3</sup>
Hexanal	237.00	J	7.76	UJ	ug/m <sup>3</sup>
HMX	1,180.00	MAX			ug/m <sup>3</sup>
Methylene Chloride	22.60		26.30	U	ppbv
m-Tolualdehyde	2.87	UJ	288.00	J	ug/m <sup>3</sup>
Nitrous Oxide	17,015.00		3,686.00		ppmv
Nonanal	5.03	UJ	34.30	UJ	ug/m <sup>3</sup>
NOx	37.10		146.20		ppmv
Octanal	2.04	UJ	13.09	UJ	ug/m <sup>3</sup>
Pentanal	2,350.00	J	1,290.00	J	ug/m <sup>3</sup>
Propanal	2.35	UJ	408.00	J	ug/m <sup>3</sup>
RDX	31,500.00				ug/m <sup>3</sup>
Total Hydrocarbons	32.80		101.20		ppmv

J = Estimated Value; concentration is below limit of quantification

MAX = Reported result was from a multi-fraction gas sampling train that contains both non-detected results and positive results

U = Analyte was not detected

### 5.2.2 Foaming:

Foaming was a problem at PANTEX during the processing of Composition B and Tetrytol explosives. The major concern other than over-flowing the reactor is trapping heat from the exothermic reaction in the hydrolysate solution. The foam is an excellent insulator and, if covering the entire liquid surface will trap heat being carried from the solution by the evolved gasses.

Foaming was encountered in all of the commissioning runs. During Run 2, the foam accumulated to a depth of nearly 12-inches and was ejected from the reactor through the reactor air vent. The water spray was inadequate to control the foaming. The foaming is attributed to the gases that are produced by hydrolysis of the HMX and RDX. The TNT decomposes into polymers forming an organic phase that is lighter than the base solution, which rises to the top of the liquid. This organic layer prevents the free flow of any gases formed from the decomposition of the RDX/HMX. The gas then forms foam as it escapes through the organic layer. This surface layer of foam can be broken-up through rapid mixing or by using an anti-foaming agent (see Pantex report, Appendix M), allowing the gas to freely escape the hydrolysate solution.

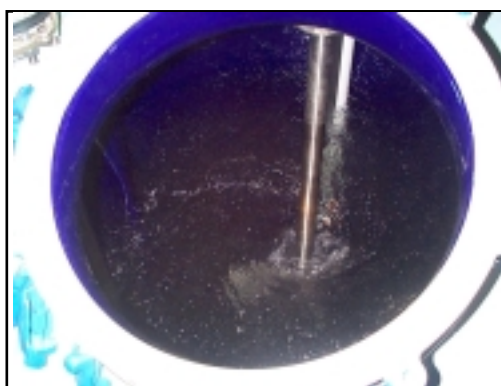


Figure 5-7. Agitation within the Hydrolysis Reactor (Vortex Formation)

The foaming was controlled during the commissioning runs by adjusting agitator speed when a build-up of foam was detected by the level sensor (the level sensor would report a level in the reactor that did not match with the calculated level based on the amount of caustic and water introduced to the reactor). Ultimately, foaming was controlled by adjusting the operating level of the hydrolysate within the reactor relative to the location of the vortex generated by the agitator blades. So long as a clearly defined vortex was maintained in the reactor, any foam formed during the hydrolysis would be quickly drawn below the surface and dissipated. Figure 5-7 shows the vortex formed by the upper agitator blade assembly (water). Controlling the level within the reactor relative to the vortex solved the problem of controlling foaming.

### 5.2.3 Overflow Incident:

On the morning after the completion of Run 2, the software defaults were accidentally “pasted over” during on-line programming that was being performed by the subcontractor. This caused the water

valve to the reactor to open and overflow the hydrolysate into the dump tank that was provided for such situation (see the FEMA). The overflow, caused by human error, was contained in the dump tank (3000-gallons of water was introduced to the reactor before the error was identified and the water valve closed).



Figure 5-8. Secondary Containment "Dump" Tank

However, some of the hydrolysate overflowed the reactor escaping through the water seal. This hydrolysate was contained by the secondary containment dike about the reactor and directed to the dump tank. This incident indicated that a packing gland seal on the agitator shaft, which would have prevented the hydrolysate from escaping the reactor, should replace the water seal. This was the only incident to occur during the commissioning or test and evaluation runs that resulted in an unintentional release of hydrolysate (confined by the secondary containment system).

### 5.3 Commissioning Runs 3 through 5.

Working at ~80% reactor volume presented problems in terms of caustic consumption and operating efficiency. Therefore, it was decided to conduct Runs 3-5 in increments as follows:

Run 3 was conducted with 20% caustic strength. Approximately 250 pounds of explosive was fed over a 2-hour period – a nominal rate of 125 lbs/hr. The starting volume was approximately 1,700-gallons.

Run 4 was conducted using the hydrolysate produced in Run 3. The adjusted caustic strength was 14%. An additional 500 pounds of Composition B explosive was fed into the hydrolysate solution at a rate of 125 lbs/hr (4-hour addition period).

Run 5 was conducted using the combined hydrolysate produced during Runs 3 and 4 as the starting solution. The caustic strength in the hydrolysate was 9.6%. An additional 500 pounds of Composition B explosive was fed into the hydrolysate solution at a rate of 500 lbs/hr.

This approach conserved caustic, minimized the amount of hydrolysis produced (waste disposal was an issue at this point in the program), maximize use of the reactor, and provide insight into a process where energetics is continuously added to a “heal” of hydrolysate of diminishing caustic strength.

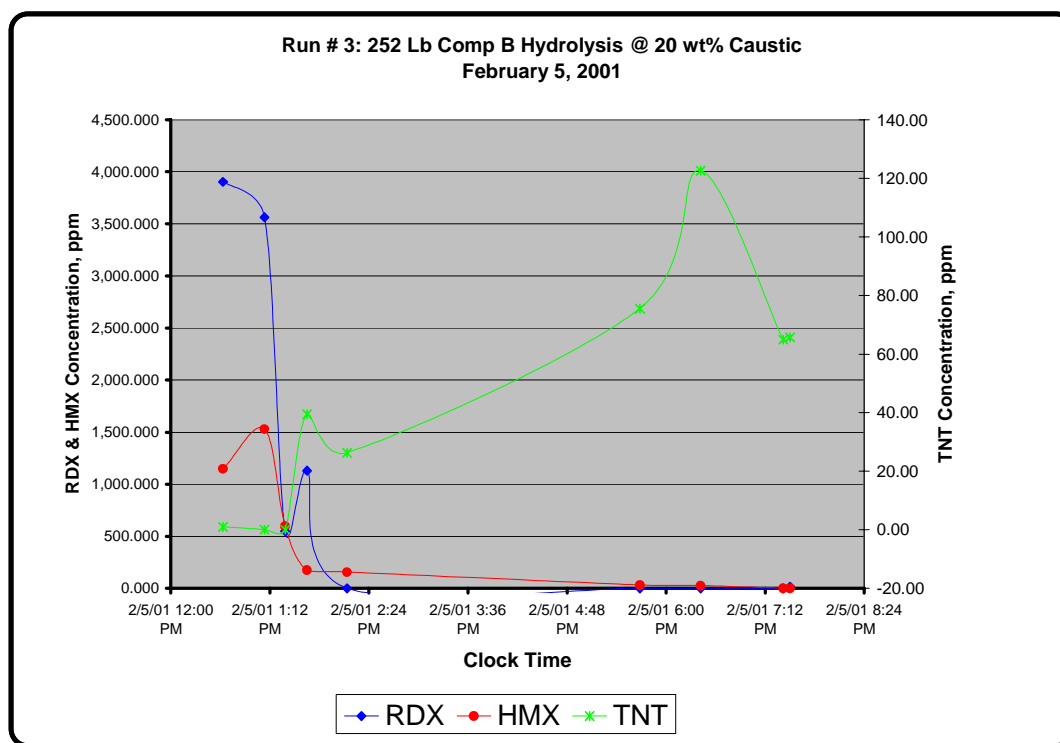


Figure 5-9. Run 3, Mid-Run Destruction Efficiency for Composition B

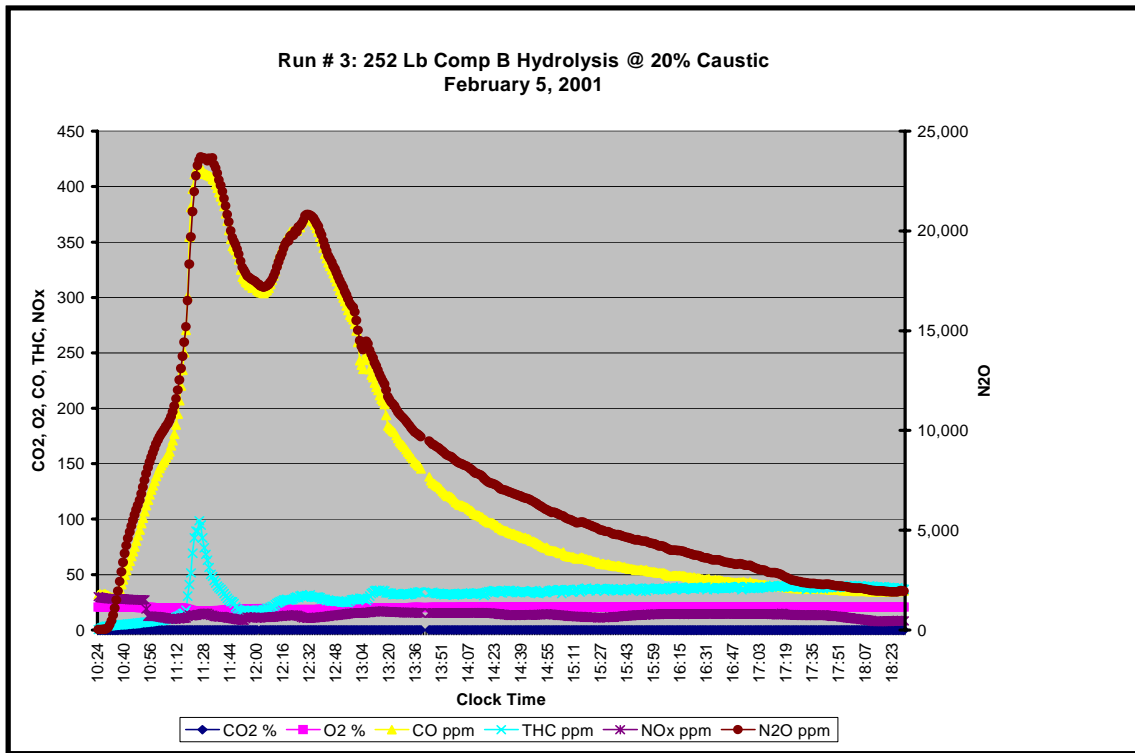


Figure 5-10. Run 3, Off-gas Production for Composition B

Table 5-3. Run 3, Composition B End of Run Hydrolysate Characterization

End of Run Hydrolysate Analysis				
Component	Concentration	Unit	ppm	Note
Acetate	1,380.00	mg/l	1,380.00	
Aluminum	9,490.00	mg/l	9,490.00	
Ammonia	1,050.00	mg/l	1,050.00	
Beryllium	2,200.00	mg/l	2,200.00	
Calcium	16,420.00	ug/l	16.42	
Chromium	12,790.00	ug/l	12.79	
Copper	90,900.00	ug/l	90.90	
Cyanide (Sodium Cyanide)	1,710.00	ug/l	1.71	
Fluoride	2,320.00	ug/l	2.32	
Formate	86,100,000.00	ug/l	86,100.00	
HMX	15,800.00	ug/l	15.80	
Iron	230.00	mg/l	230.00	J
Lead	5.90	ug/l	0.01	J
Magnesium	1,000.00	ug/l	1.00	J
Mercury	2.20	ug/l	0.00	J
Nitrite-N	13,000.00	ug/l	13.00	J
Sodium	210.00	ug/l	0.21	J
TNT	430.00	ug/l	0.43	J
Zinc	3,700.00	ug/l	3.70	J
TIC	684.25	mg/l	684.25	
TOC	7,171.00	mg/l	7,171.00	
COD	18,900.00	mg/l	18,900.00	
Total Suspended Solids	840.00	mg/l	840.00	J
Total Dissolved Solids	201,000.00	mg/l	201,000.00	
Normality as NaOH	7.50	n		
Density	1.15	g/ml		

J = Estimated Value; concentration is below limit of quantification

Table 5-4. Run 3, Composition B Off-gas Analysis

Reactor Off Gas Analysis					
Component	During Energetic Addition	Note	During Reaction	Note	Unit
2,4,6-Trinitrotoluene	3,370.00	MAX			ug/m <sup>3</sup>
2,4-Dinitrotoluene	88.60	MAX			ug/m <sup>3</sup>
2,6-Dinitrotoluene	31.90	MAX			ug/m <sup>3</sup>
2-Amino-4,6-Dinitrotoluene	119.00	MAX			ug/m <sup>3</sup>
4-Amino-2,6-Dinitrotoluene	150.00	MAX			ug/m <sup>3</sup>
Acetaldehyde	311.00		20.60		ug/m <sup>3</sup>
Acetone	414.00		1,140.00		ppbv
Ammonia	3,570,000.00		4,920,000.00		ug/m <sup>3</sup>
Butanal	43.60		15.30		ug/m <sup>3</sup>
Carbon Dioxide	0.09		0.05		%
Carbon Disulfide	26.80		35.20		ppbv
Carbon Monoxide	216.00		62.00		ppmv
Chloroform	27.80				ppbv
Crotonaldehyde	4.71	J	0.86	J	ug/m <sup>3</sup>
Cyclohexanone	1,760.00		31.60		ug/m <sup>3</sup>
Decanal	387.00		40.90		ug/m <sup>3</sup>
Dibromochloromethane	12.50		10.00	U	ppbv
Formaldehyde	3,590.00	D	144.00		ug/m <sup>3</sup>
Heptanal	29.40		1.12	U	ug/m <sup>3</sup>
Hexanal	39.80		2.84	J	ug/m <sup>3</sup>
HMX	28.90	MAX			ug/m <sup>3</sup>
Methylene Chloride	54.30	B	113.00	B	ppbv
m-Tolualdehyde	13.80				ug/m <sup>3</sup>
Nitrous Oxide	13,875.00		6,784.00		ppmv
Nonanal	32.50				ug/m <sup>3</sup>
NOx	0.00		0.00		ppmv
Octanal	37.10				ug/m <sup>3</sup>
Oxygen	19.50		20.70		%
Propanal	288.00		24.80		ug/m <sup>3</sup>
RDX	2,220.00	MAX			ug/m <sup>3</sup>
Toluene			12.50		ppbv
Total Hydrocarbons	31.00		45.10		ppmv

J = Estimated Value; concentration is below limit of quantification

MAX = Reported result was from a multi-fraction gas sampling train that contains both non-detected results and positive results

U = Analyte was not detected

D = Result was obtained from analysis of a dilution or surrogate were diluted below detection limit

B = When applied to anions or organic analysis the qualifier indicates that the analyte was detected in the associated method/instrument blank



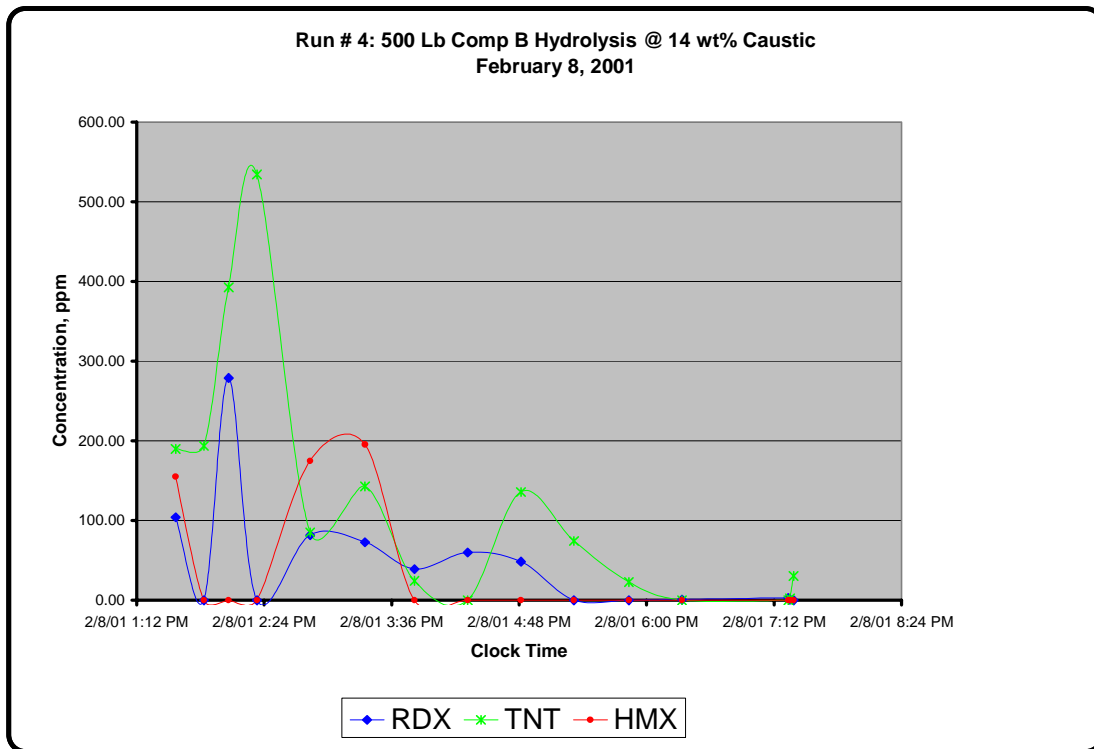


Figure 5-11. Run 4, Mid-Run Destruction Efficiency for Composition B

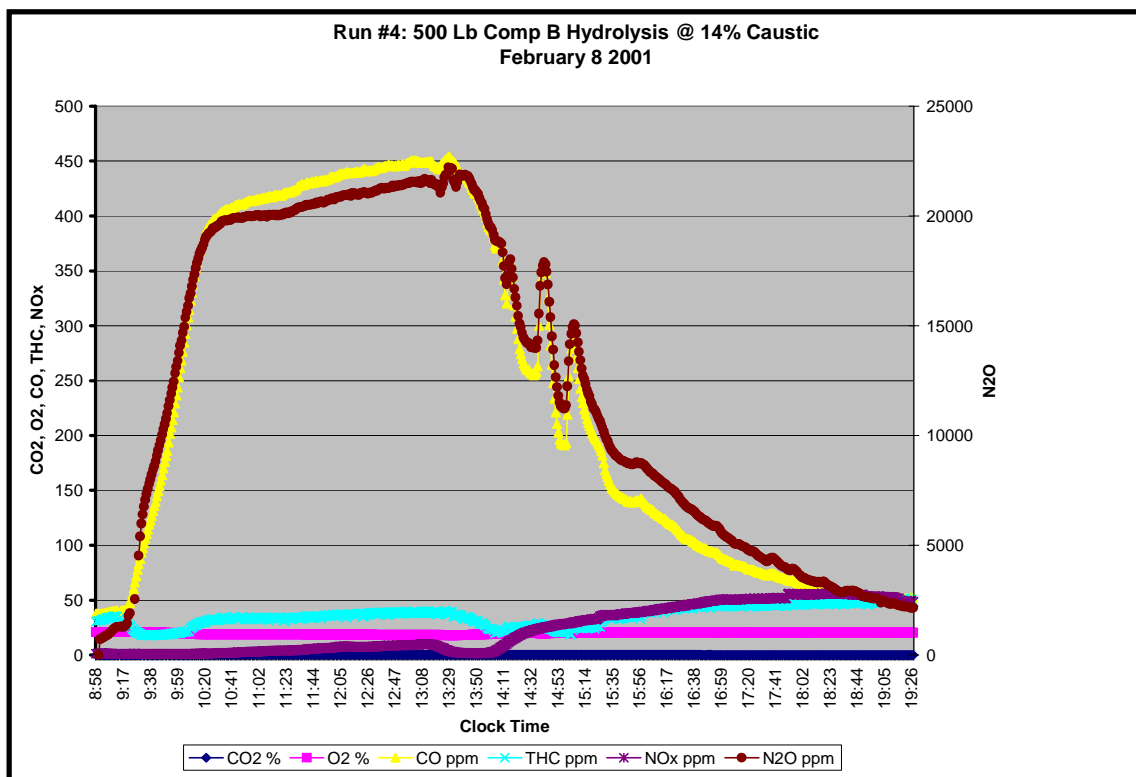


Figure 5-12. Run 4, Off-gas Production for Composition B

Table 5-5. Run 4, Composition B End of Run Hydrolysate Characterization

End of Run Hydrolysate Analysis				
Component	Concentration	Unit	ppm	Note
Acetate	3,010.00	mg/l	3,010.00	
Aluminum	960.00	ug/l	0.96	J
Ammonia	2,540.00	mg/l	2,540.00	
Beryllium	2.60	ug/l	0.0026	J
Calcium	16,000.00	ug/l	16	J
Chromium	230.00	ug/l	0.23	J
Cobalt	150.00	ug/l	0.15	J
Copper	440.00	ug/l	0.44	J
Cyanide (Sodium Cyanide)	40,100.00	ug/l	40.1	
Fluoride	260.00	mg/l	260.00	J
Formate	20,200.00	mg/l	20,200.00	
Iron	3,000.00	ug/l	3	J
Lead	530.00	ug/l	0.53	J
Magnesium	4,210.00	ug/l	4.21	
Manganese	69.00	ug/l	0.069	J
Nitrite-N	5,100.00	mg/l	5,100.00	
Phosphorus	530.00	ug/l	0.53	J
Potassium	22,000.00	ug/l	22	J
Silver	59.00	ug/l	0.059	J
Sodium	68,900,000.00	ug/l	68900	
TNT	2,720.00	ug/l	2.72	
Zinc	8,980.00	ug/l	8.98	
TIC	1,380.00	mg/l	1,380.00	
TOC	17,537.50	mg/l	17,537.50	
COD	41,400.00	mg/l	41,400.00	
Total Dissolved Solids	187,000.00	mg/l	187,000.00	
Total Suspended Solids	164.00	mg/l	164.00	
Normality as NaOH	4.25	n		
Density	1.12	g/ml		

J = Estimated Value; concentration is below limit of quantification

Table 5-6. Run 4, Composition B Off-gas Analysis

Reactor Off Gas Analysis					
Component	During Energetic Addition	Note	During Reaction	Note	Unit
1,3,5- Trinitrobenzene	37.30	MAX			ug/m <sup>3</sup>
1,3-Dinitrobenzene	3.65	MAX			ug/m <sup>3</sup>
2,4,6-Trinitrotoluene	6710.00	MAX			ug/m <sup>3</sup>
2,4-Dinitrotoluene	124.00	MAX			ug/m <sup>3</sup>
2,6-Dinitrotoluene	33.60	MAX			ug/m <sup>3</sup>
2-Amino-4,6-Dinitrotoluene	37.30	MAX			ug/m <sup>3</sup>
4-Amino-2,6-Dinitrotoluene	57.40	MAX			ug/m <sup>3</sup>
Acetaldehyde	1350.00		69.10		ug/m <sup>3</sup>
Acetone	552.00		404.00		ppbv
Ammonia	4110000.00		16,200,000.00		ug/m <sup>3</sup>
Bromodichloroethane	20.00		13.80	U	ppbv
Butanal	87.70		29.70		ug/m <sup>3</sup>
Carbon Dioxide	0.12		0.07		%
Carbon Monoxide	323.00		123.00		ppmv
Chloroform	16.10		13.80	U	ppbv
Crotonaldehyde	14.80		0.56	U	ug/m <sup>3</sup>
Cyanide	0.01		0.01		ug/m <sup>3</sup>
Cyclohexanone	6260.00	D	278.00		ug/m <sup>3</sup>
Decanal	619.00		175.00		ug/m <sup>3</sup>
Dibromochloromethane	20.50		13.80	U	ppbv
Formaldehyde	6870.00	D	347.00		ug/m <sup>3</sup>
Heptanal	34.60		21.00		ug/m <sup>3</sup>
Hexanal	40.00		29.10		ug/m <sup>3</sup>
HMX	16.20	MAX			ug/m <sup>3</sup>
Methylene Chloride	73.30	B	90.90	B	ppbv
m-Tolualdehyde	0.36	J	13.00		ug/m <sup>3</sup>
Nitrous Oxide	18089.00		9,180.00		ppmv
Nonanal	47.10		22.60		ug/m <sup>3</sup>
NOx	0.00		16.80		ppmv
Octanal	50.10		23.80		ug/m <sup>3</sup>
Oxygen	18.90		20.00		%
Propanal	454.00		83.30		ug/m <sup>3</sup>
RDX	3,690.00	MAX			ug/m <sup>3</sup>
Toluene	12.70		13.80	U	ppbv
Total Hydrocarbons	42.60		47.90		ppmv

J = Estimated Value; concentration is below limit of quantification

MAX = Reported result was from a multi-fraction gas sampling train that contains both non-detected results and positive results

U = Analyte was not detected

D = Result was obtained from analysis of a dilution or surrogate were diluted below detection limit

B = When applied to anions or organic analysis the qualifier indicates that the analyte was detected in the associated method/instrument blank

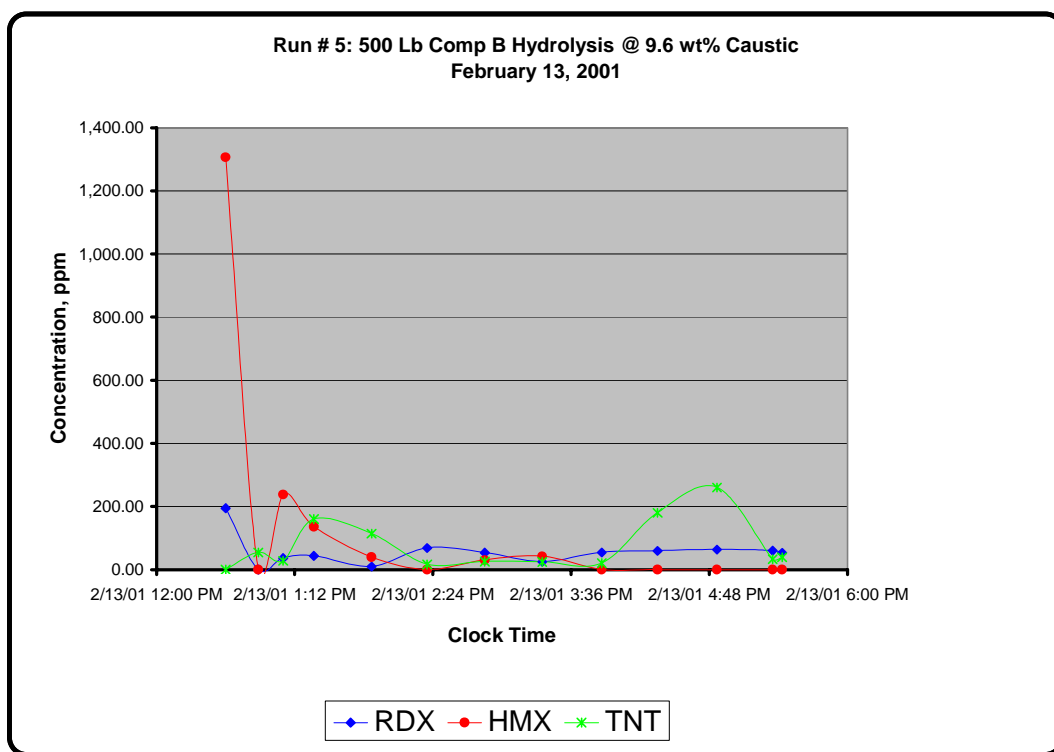


Figure 5-13. Run 5, Mid-Run Destruction Efficiency for Composition B

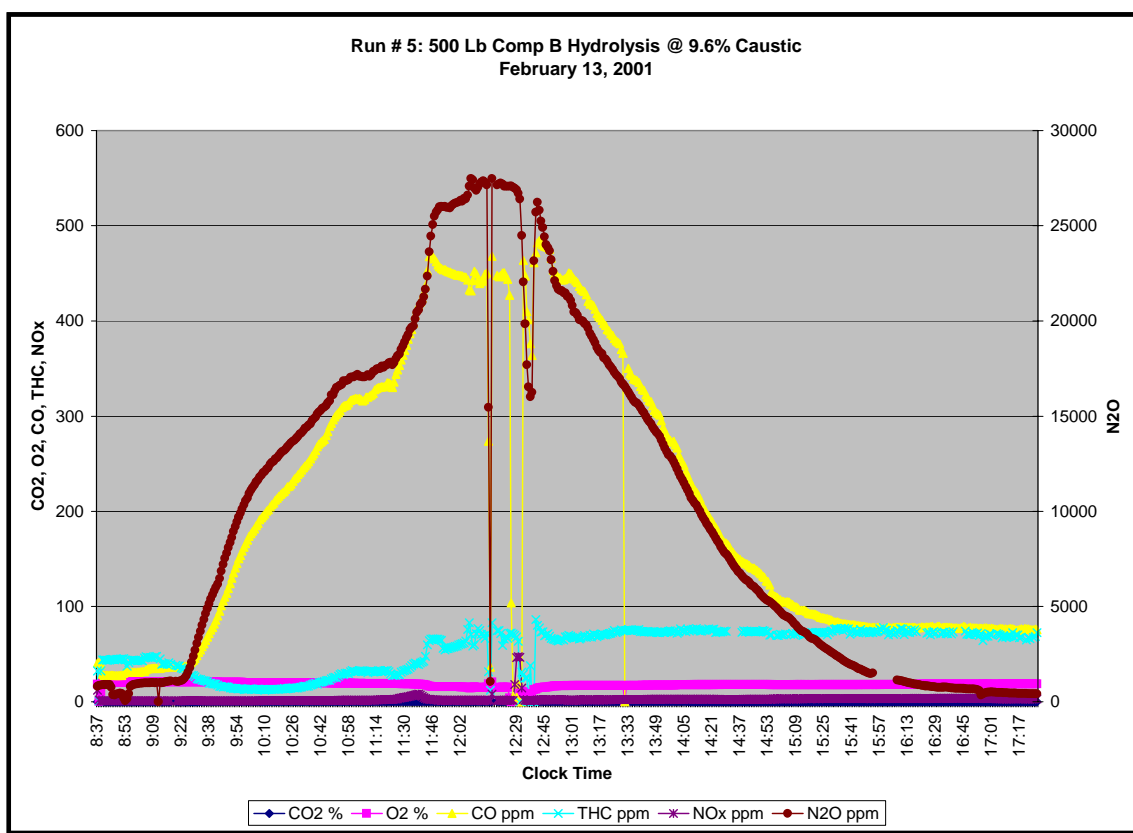


Figure 5-14. Run 5, Off-gas Production for Composition

Table 5-7. Run 5, Composition B End of Run Hydrolysate Characterization

End of Run Hydrolysate Analysis				
Component	Concentration	Unit	ppm	Note
Acetate	3,680.00	mg/l	3,680.00	
Aluminum	1300	ug/l	1.30	J
Ammonia	1,380.00	mg/l	1,380.00	
Beryllium	3.8	ug/l	0.00	J
Calcium	24000	ug/l	24.00	J
Chromium	160	ug/l	0.16	J
Cobalt	200	ug/l	0.20	J
Copper	380	ug/l	0.38	J
Cyanide (Sodium Cyanide)	40,000.00	ug/l	40.00	
Formate	27,600.00	mg/l	27,600.00	
Iron	2700	ug/l	2.70	J
Lead	670	ug/l	0.67	J
Magnesium	5,920.00	ug/l	5.92	
Nitrite-N	123.00	mg/l	123.00	
Silver	85	ug/l	0.09	J
Sodium	62,200,000.00	ug/l	62,200.00	
Sulfate	149.00	mg/l	149.00	
TNT	24,940.00	ug/l	24.94	
Zinc	3,880.00	ug/l	3.88	
TIC	1,917.50	mg/l	1,917.50	
TOC	21,190.00	mg/l	21,190.00	
COD	56,000.00	mg/l	56,000.00	
Total Suspended Solids	170.00	mg/l	170.00	
Total Dissolved Solids	176,000.00	mg/l	176,000.00	
Normality as NaOH	1.15	n		
Density	1.12	g/ml		

J = Estimated Value; concentration is below limit of quantification

Table 5-8. Run 5, Composition B Off-gas Analysis

Reactor Off Gas Analysis					
Component	During Energetic Addition	Note	During Reaction	Note	Unit
1,1,2,2-Tetrachloroethane	6.00	U	8.80		ppbv
1,3,5- Trinitrobenzene	7.14	MAX			ug/m <sup>3</sup>
1,3-Dinitrobenzene	1.61	MAX			ug/m <sup>3</sup>
2,4,6-Trinitrotoluene	1,710.00	MAX			ug/m <sup>3</sup>
2,4-Dinitrotoluene	40.80	MAX			ug/m <sup>3</sup>
2,6-Dinitrotoluene	10.70	MAX			ug/m <sup>3</sup>
2-Amino-4,6-Dinitrotoluene	23.20	MAX			ug/m <sup>3</sup>
4-Amino-2,6-Dinitrotoluene	33.30	MAX			ug/m <sup>3</sup>
Acetaldehyde	442.00		78.40		ug/m <sup>3</sup>
Acetone	6.00	U	262.00		ppbv
Acetonitrile	482.50	J	4,651.15	J	ppbv
Ammonia	8,780,000.00		82,300,000.00		ug/m <sup>3</sup>
Benzene	6.00	U	33.30		ppbv
Bromodichloroethane	28.70		8.80	U	ppbv
Butanal	46.70		42.80		ug/m <sup>3</sup>
Carbon Dioxide	0.09		0.04		%
Carbon Monoxide	238.00		153.00		ppmv
Chloroform	23.60		8.80	U	ppbv
Crotonaldehyde	1.01	J	0.78	U	ug/m <sup>3</sup>
Cyanide	0.00	U	0.04		ug/m <sup>3</sup>
Cyclohexanone	3,710.00		33.10		ug/m <sup>3</sup>
Decanal	164.00		44.90		ug/m <sup>3</sup>
Dibromochloromethane	27.00		8.80	U	ppbv
Formaldehyde	1,720.00		142.00		ug/m <sup>3</sup>
HMX	9.25	MAX			ug/m <sup>3</sup>
Methylene Chloride	44.30	B	97.20	B	ppbv
m-Tolualdehyde	0.89	J	8.30		ug/m <sup>3</sup>
Nitrous Oxide	15459.00		9,078.00		ppmv
Nonanal	51.20		31.40		ug/m <sup>3</sup>
NOx	1.30		2.30		ppmv
Octanal	52.50		37.00		ug/m <sup>3</sup>
Oxygen	19.20		18.00		%
RDX	403.00	MAX			ug/m <sup>3</sup>
Silanol, trimethyl-			221.79	J	ppbv
Toluene	4.74		8.80	U	ppbv
Total Hydrocarbons	40.70		113.80		ppmv

J = Estimated Value; concentration is below limit of quantification

MAX = Reported result was from a multi-fraction gas sampling train that contains both non-detected results and positive results

U = Analyte was not detected

D = Result was obtained from analysis of a dilution or surrogate were diluted below detection limit

B = When applied to anions or organic analysis the qualifier indicates that the analyte was detected in the associated method/instrument blank

### 5.3.1 Spray Nozzle:

The original Teflon spray nozzle with rotating head provided by Pfaudler to control dusting during the energetics addition did not perform very well; the water droplets generated were too large to be effective and the flow rate was too high (about 20 gpm), adding a significant volume of water to the hydrolysate and raising the water level within the reactor. The spray nozzle was used sparingly during Runs 3 through 5, and the spray nozzle assembly was redesigned before proceeding with the test runs.

RONA's solution for the spray nozzle was to fabricate a 1.5-inch diameter stainless steel pipe, which was welded closed on the end and equipped with three fixed spray nozzles (( McMaster Carr Catalog 106 (Stock # 32815K52) on the outside of the pipe. Each nozzle had a flow rate of 0.38 gpm, which correlated to an approximate total flow rate of 1.2 gpm. The nozzles were very effective in providing a "fine mist" spray. The nozzles were oriented to provide a "horizontal" stream of the mist spray that covered the entire surface of the vessel. In other words, a blanket or cloud of mist would cover the surface of the hydrolysate (and in some cases foam). The resulting droplets hitting the surface of the hydrolysate were effective in "breaking up" and controlling the foam (when combined with high agitation, which created a reasonably strong vortex).

### 5.3.2 Discussion of Commissioning Results:

The commissioning runs indicated that the system could be operated safely and efficiently, and that the formal test program could commence. Many equipment improvements were identified that would be implemented as the program proceeded (or at a later date) with the caveat that these improvements did not delay the test schedule. Some of the areas identified included:

- Replace agitator water seal with a packing gland or rotary seal
- Develop a software logic to link the level detector output to water and caustic totalizers to simplify the identification of foaming
- Improved NO scrubbing capabilities including auto-emissions detection linked to the PC software
- Heat exchanger programming for accurate temperature control of the set point
- Water and caustic preheating to shorten start-up time
- Feeding of energetics in slurry form to increase throughput and simplify handling

The results of the liquid analyses performed at the Holston AAP laboratory confirmed that the Composition B explosive was destroyed to below the DRE goal of 99.999%.

The ups and downs in the concentration of the energetics during mid run sampling and analysis for Runs 3 through 5 is most likely due to the fact that some energetic particles are entering and passing through the recycle loop where the samples are being collected by the auto-sampler system. The important thing is that at the end of the run there are no energetic materials in the hydrolysate and if there are some, the concentration is very low. Please note: it is not easy to draw a liquid sample from a 2,000-gallon dynamic reactor and have a representative sample of the reactor content.

During the off gas stream characterization of Runs 3 through 5, it was revealed that some energetic materials (TNT, DNT, RDX, HMX, 1,3,5 - Trinitrobenzene) are present during the addition of energetics and disappeared once addition is completed. TNT and DNT have measurable vapor pressures at ambient temperatures; therefore, one would expect these materials to be included in the off-gassing stream. The presence of RDX and HMX in the off gas stream is most likely contributed to the fact these materials are entrained in water droplets in the off gas stream. Also, some dusting occurs as the energetic materials fall into the reactor through the headspace air stream, possibly contributing to these readings. Use of a condenser, just above the reactor, and a water-mister to drop any entrained materials back into the reactor should be effective in confining these materials to within the reactor system.

The inorganic materials (metals) detected in the hydrolysate end of runs analysis are sourced from the sodium hydroxide stock feed that contains some of these components.

## 6.0 ENERGETICS HYDROLYSIS TESTS

The tests conditions for the following energetics were established to determine the optimum caustic soda concentration, which will insure maximum destruction efficiency (< 99.999%) at a minimum reactor residence time.

<u>Composition B4 Explosive</u>	Nominal composition is 59.75% RDX (includes HMX percentages varying from 5-20%), 39.75% TNT, and 0.50% calcium silicate
<u>M1 Propellant</u>	Nominal composition is 84% nitrocellulose, 9% Dinitrotoluene, 5% dibutylphthalate, 1% diphenylamine, and 1% lead carbonate
<u>M8 propellant (sheet)</u>	Nominal composition is 52.15% nitrocellulose, 43% nitroglycerin, 3% diethylphthalate, 1.25% potassium nitrate, and 0.60% ethyl centralite
<u>Tetrytol Explosive</u>	Nominal composition is 70% Tetryl and 30% TNT
<u>M28 Propellant (granular)</u>	Nominal composition is 60% nitrocellulose, 23.8% nitroglycerin, 9.9% triacetin, 2.6% dimethylphthalate, and 2.0% lead stearate, and 1.7% 2-nitrodiphenylamine

In addition, a mixture of M28 leaded propellant and Composition B4 explosive (86/14 weight-percent based on their amount/ratio in the 115-mm Rocket, Chemical Agent VX, M55) was hydrolyzed to address NRC concerns.

It is also the objective of these runs to fully characterize the hydrolysate by-product and off-gas during energetic hydrolysis and at the end of the each run.

The tests were conducted at 12-, 20- and 25-weight percent caustic strength and at a nominal feed rate of: 1<sup>st</sup> hour - 50 lbs. hour; 2<sup>nd</sup> hour - 100 lbs. per hour; 3<sup>rd</sup> hour - 150 lbs. per hour; 4<sup>th</sup> hour - 200 lbs. per hour for a total processed weight of 500 pounds (unless otherwise stated in the respective tables).

To minimize the amount of caustic consumed during the tests, the operating level within the reactor was set just above the lower agitator blade; a volume equivalent to approximately 700-gallons.

Tests were not combined (i.e., the hydrolysate from one test was not carried over to the next test) to ensure that the liquid analyses and off-gassing profiles were unique to the respective caustic solution strength, and so as not to have residuals compounds from a previous test possibly effect the results of the current test. The tests were structured in this manner to examine the effect the various feed rates and caustic strengths had on evolved gasses, rate of reaction, by-products, etc.

The hydrolysate analysis and data collection was performed after completion of the 4-hour addition time unless otherwise specified. The off-gas analysis and data collection was performed from the beginning of the run.

The Test Plan Requirements are provided in Appendix N.



## 6.1 Composition B4 Explosive Hydrolysis Tests & Results (Runs 6, 7, & 8).

The objective of these tests is to clearly determine and define the optimum operating parameters for the Composition B and Composition B4 explosives hydrolysis process to support the design and installation of the full-scale hydrolysis process at the Blue Grass Chemical Agent Disposal Facility for the destruction of the explosives contained in the 8-inch Projectile, Chemical Agent GB, M42; 155mm Rocket, Chemical Agent GB, M55; 155mm Projectile, Chemical Agent VX, M121/A1; 155mm Rocket, Chemical Agent VX, M55 and 155mm Rocket Warhead, Chemical Agent VX, M56.

The table below identifies the process operating parameters for Runs 6, 7, and 8.

Table 6-1. Composition B4 Explosive Test Parameters

Operating Condition	Run 6	Run 7	Run 8
Composition B4 Feed Rate, lbs/hr			
1 <sup>st</sup> hour	50	50	50
2 <sup>nd</sup> hour	100	100	100
3 <sup>rd</sup> hour	150	150	150
4th hour	200	200	200
Caustic Soda Concentration, wt%	12	18	25
Caustic Soda Feed, gal	700	700	700
Reactor Operating Temperature, °C	87	87	87
Agitation Speed, RPM	70	70	70
Date Conducted	2/15/2001	2/20/2001	2/26/2001

The following charts present the process operating and control parameters for hydrolysis of Composition B4 explosive for Runs 6 and 8. The chart for Run 7 is not included because the data logger failed to record the proper time sequence.

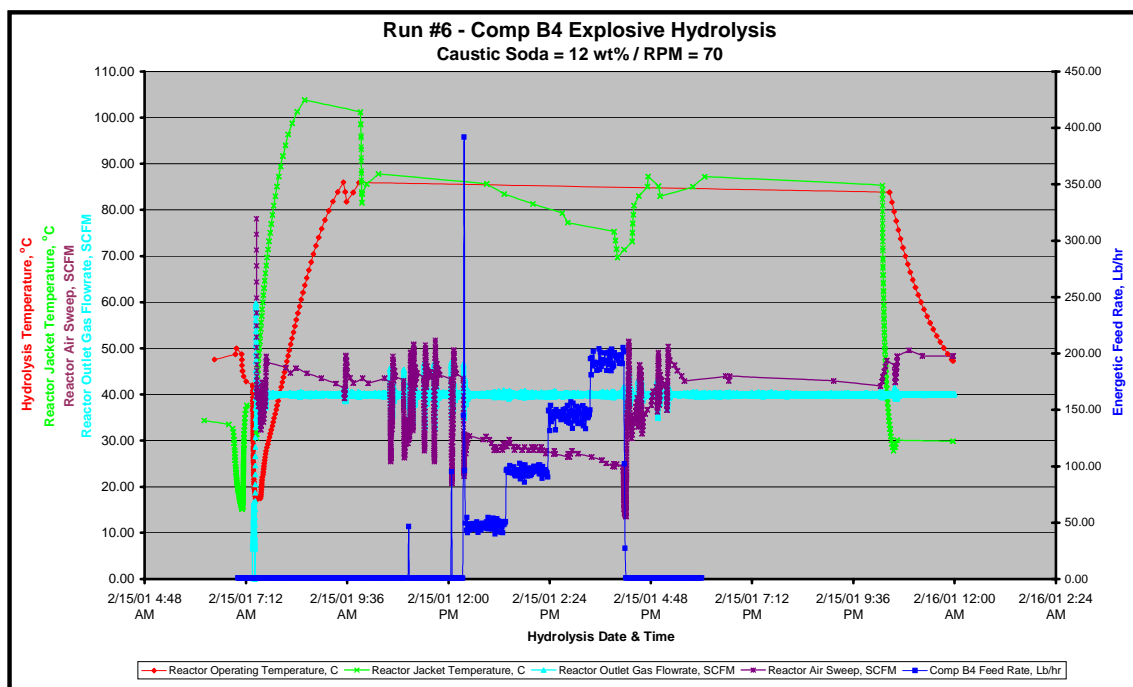


Figure 6-1. Run 6, Process Operating and Control Parameters

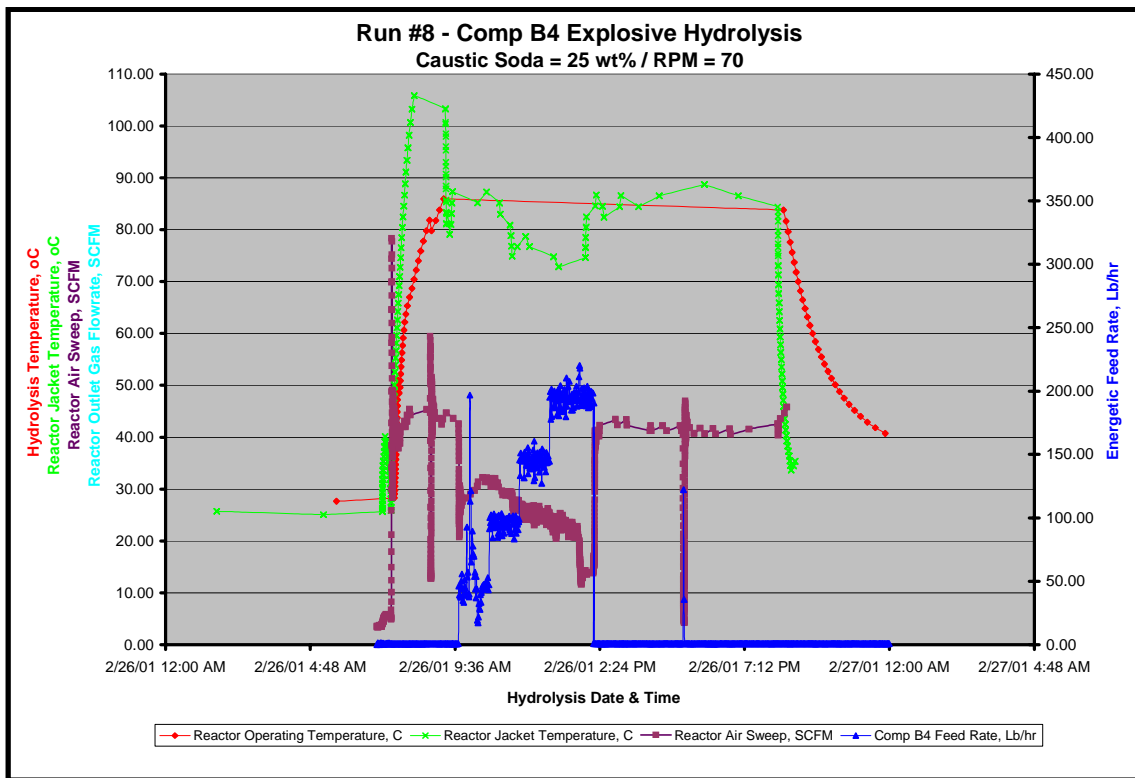


Figure 6-2. Run 8, Process Operating and Control Parameters

#### 6.1.1 Tests Results:

The plots present Composition B4 explosive destruction as a function of reactor residence time.

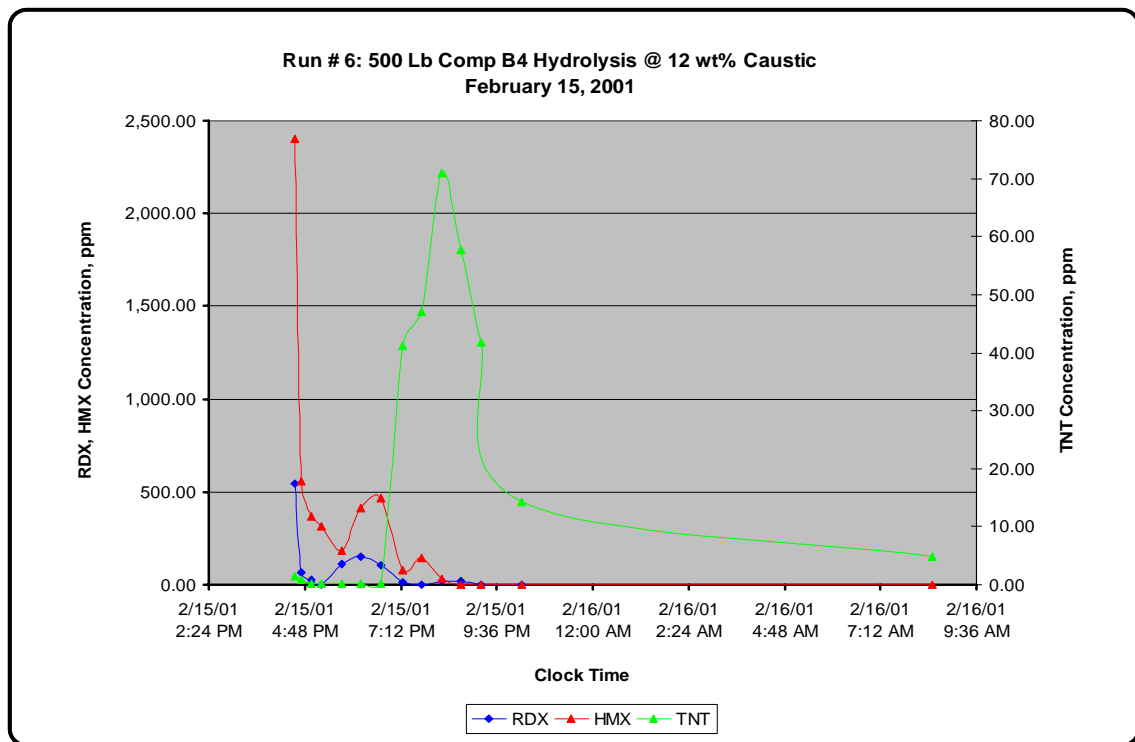


Figure 6-3. Run 6, Mid-Run Destruction Efficiency

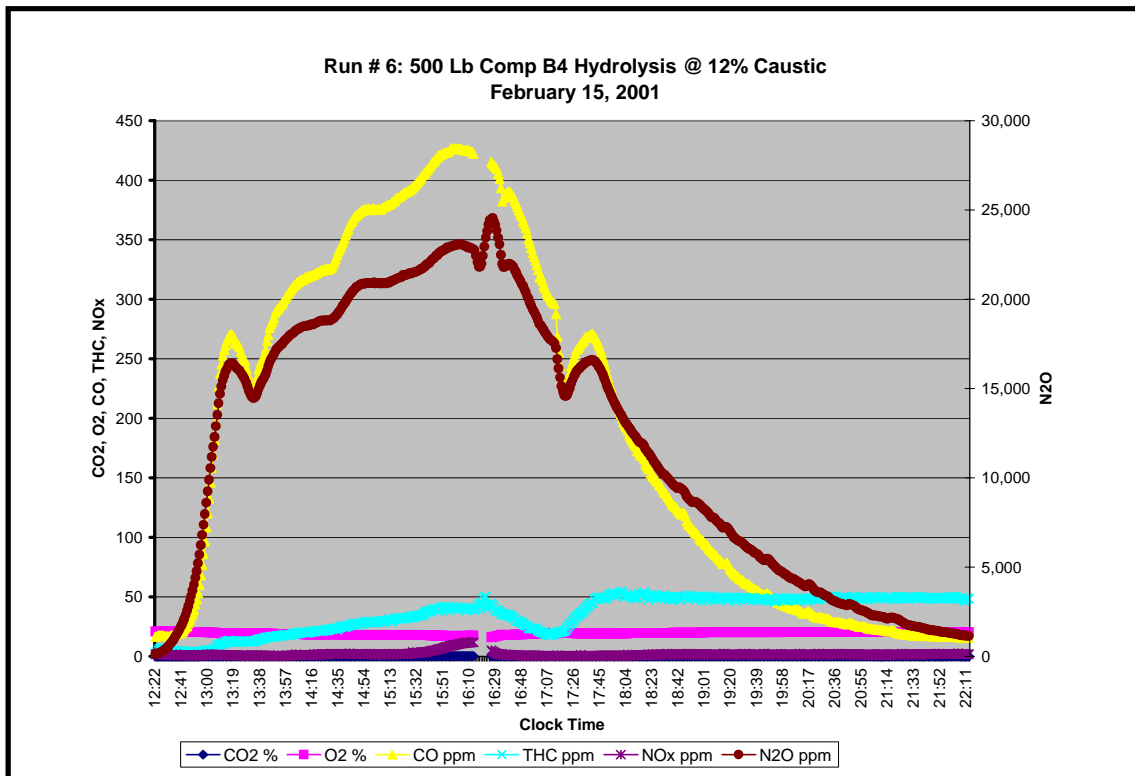


Figure 6-4. Run 6, Off-gas Production

Table 6-2. Run 6, Composition B4 End of Run Hydrolysate Characterization

End of Run Hydrolysate Analysis				
Component	Concentration	Unit	ppm	Note
Acetate	1,700.00	mg/l	1,700.00	
Aluminum	3,620.00	ug/l	3.62	
Ammonia	1,720.00	mg/l	1,720.00	
Calcium	90,300.00	ug/l	90.30	
Chloride	370.00	mg/l	370.00	J
Chromium	120.00	ug/l	0.12	J
Cobalt	100.00	ug/l	0.10	J
Copper	1,300.00	ug/l	1.30	
Cyanide (Sodium Cyanide)	39,200.00	ug/l	39.20	
Formate	10,900.00	mg/l	10,900.00	
Iron	3,700.00	ug/l	3.70	J
Lead	380.00	ug/l	0.38	J
Magnesium	7,120.00	ug/l	7.12	
Mercury	1.20	ug/l	0.00	J
Nitrite-N	3,800.00	mg/l	3,800.00	
Sodium	53,300,000.00	ug/l	53,300.00	
Sulfate	92.00	mg/l	92.00	J
TNT	4,890.00	ug/l	4.89	
Zinc	6,830.00	ug/l	6.83	
TIC	983.50	mg/l	983.50	
TOC	13,053.33	mg/l	13,053.33	
COD	30,800.00	mg/l	30,800.00	
Total Dissolved Solids	141,000.00	mg/l	141,000.00	
Total Suspended Solids	308.00	mg/l	308.00	
Normality as NaOH	1.64	n		
Density	1.09	g/ml		

J = Estimated Value; concentration is below limit of quantification

Table 6-3. Run 6, Composition B4 Off-gas Analysis

Reactor Off Gas Analysis					
Component	During Energetic Addition	Note	During Reaction	Note	Unit
1,3,5-Trinitrobenzene	28.60	MAX			ug/m <sup>3</sup>
1,3-Dinitrobenzene	6.29	MAX			ug/m <sup>3</sup>
2,4,6-Trinitrotoluene	6,940.00	MAX			ug/m <sup>3</sup>
2,4-Dinitrotoluene	165.00	MAX			ug/m <sup>3</sup>
2,6-Dinitrotoluene	37.20	MAX			ug/m <sup>3</sup>
2-Amino-4,6-Dinitrotoluene	34.40	MAX			ug/m <sup>3</sup>
4-Amino-2,6-Dinitrotoluene	51.60	MAX			ug/m <sup>3</sup>
Acetaldehyde	1,060.00		41.50		ug/m <sup>3</sup>
Acetone	555.00		103.00		ppbv
Acetonitrile	1,952.16	J			ppbv
Ammonia	5,030,000.00		16,900,000.00		ug/m <sup>3</sup>
Benzene	17.70				ppbv
Bromodichloromethane	26.80				ppbv
Bromoform	16.50				ppbv
Butanal	36.00		13.00		ug/m <sup>3</sup>
Carbon Dioxide	0.08		0.05		%
Carbon Monoxide	284.00		112.00		ppmv
Chloroform	15.30				ppbv
Crotonaldehyde	9.71		2.37	J	ug/m <sup>3</sup>
Cyanide	0.01		0.01		ug/m <sup>3</sup>
Cyclohexanone			396.00		ug/m <sup>3</sup>
Decanal	1,170.00		91.00		ug/m <sup>3</sup>
Dibromochloromethane	35.60				ppbv
Formaldehyde			807.00		ug/m <sup>3</sup>
Heptanal	71.70		23.30		ug/m <sup>3</sup>
Hexanal	279.00		4.98	J	ug/m <sup>3</sup>
HMX	10.00	MAX			ug/m <sup>3</sup>
Isopropyl Alcohol			260.82	J	ppbv
m-Tolualdehyde	99.10		10.40		ug/m <sup>3</sup>
Nitrous Oxide	16,394.00		9,231.00		ppmv
Nonanal	60.10				ug/m <sup>3</sup>
NOx	0.90		0.00		ppmv
Octanal	63.50		23.40		ug/m <sup>3</sup>
Oxygen	19.00		20.20		%
Pentanal	6.30	J			ug/m <sup>3</sup>
Propanal	533.00		21.30		ug/m <sup>3</sup>
RDX	264.00	MAX			ug/m <sup>3</sup>
Total Hydrocarbons	27.60		70.80		ppmv

J = Estimated Value; concentration is below limit of quantification

MAX = Reported result was from a multi-fraction gas sampling train that contains both non-detected results and positive results

U = Analyte was not detected

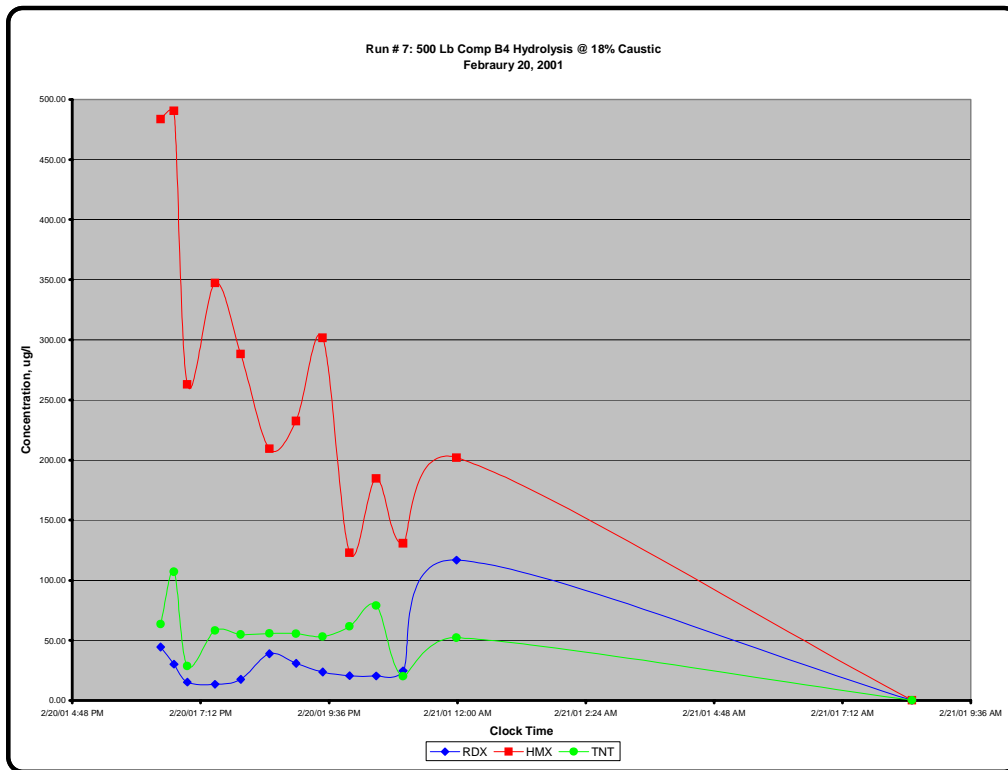


Figure 6-5. Run 7, Mid-Run Destruction Efficiency

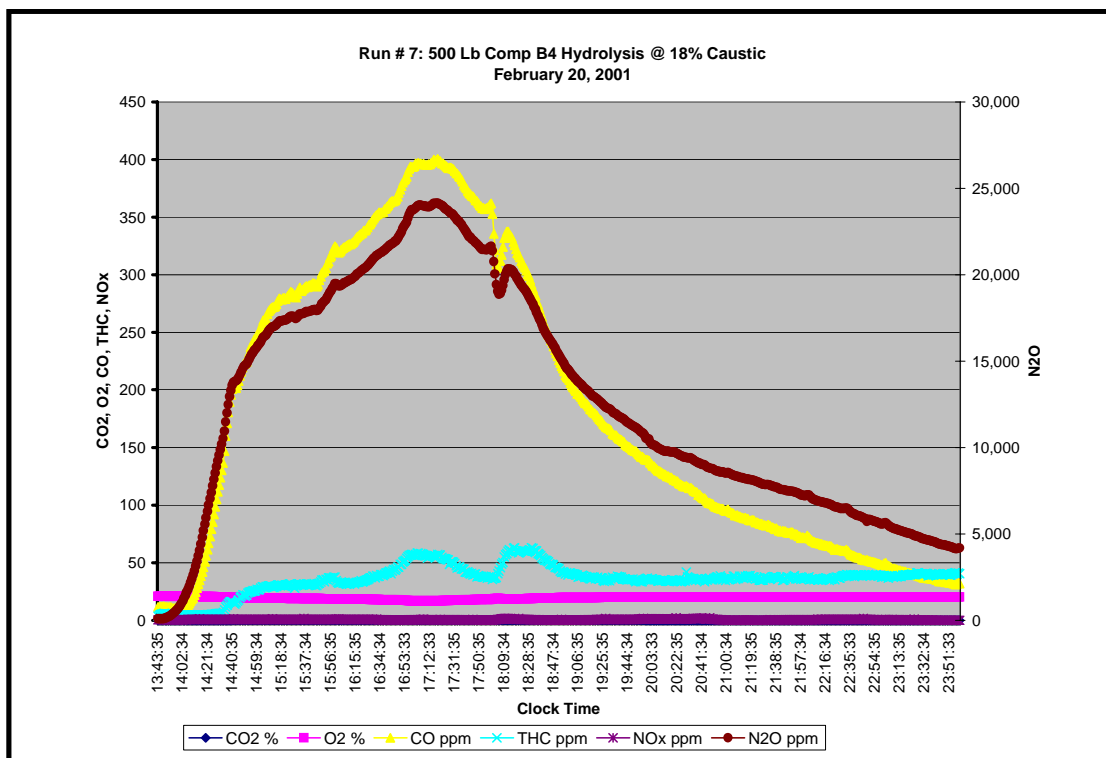


Figure 6-6. Run 7, Off-gas Production

Table 6-4. Run 7, Composition B4 End of Run Hydrolysate Characterization

End of Run Hydrolysate Analysis				
Component	Concentration	Unit	ppm	Note
Acetate	2,120.00	mg/l	2,120.00	
Aluminum	3,400.00	ug/l	3.40	
Ammonia	1,760.00	mg/l	1,760.00	
Calcium	70,600.00	ug/l	70.60	
Chloride	390.00	mg/l	390.00	J
Chromium	140.00	ug/l	0.14	J
Cobalt	110.00	ug/l	0.11	J
Copper	1,670.00	ug/l	1.67	
Cyanide (Sodium Cyanide)	59,200.00	ug/l	59.20	
Fluoride	96.00	mg/l	96.00	J
Formate	16,200.00	mg/l	16,200.00	
Iron	5,100.00	ug/l	5.10	J
Lead	330.00	ug/l	0.33	J
Magnesium	2,430.00	ug/l	2.43	
Molybdenum	110.00	ug/l	0.11	J
Nitrite-N	4,300.00	mg/l	4,300.00	
Potassium	84,000.00	ug/l	84.00	J
Sodium	84,800,000.00	ug/l	84,800.00	
TNT	4,890.00	ug/l	4.89	
Zinc	1,250.00	ug/l	1.25	
TIC	1,090.00	mg/l	1,090.00	
TOC	16,960.00	mg/l	16,960.00	
COD	34,300.00	mg/l	34,300.00	
Total Dissolved Solids	214,000.00	mg/l	214,000.00	
Total Suspended Solids	500.00	mg/l	500.00	
Normality as NaOH	3.00	n		
Density	1.17	g/ml		

J = Estimated Value; concentration is below limit of quantification

Table 6-5. Run 7, Composition B4 Off-gas Characterization

Reactor Off Gas Analysis					
Component	During Energetic Addition	Note	During Reaction	Note	Unit
1,3,5-Trinitrobenzene	21.90	MAX			ug/m <sup>3</sup>
1,3-Dinitrobenzene	6.89	MAX			ug/m <sup>3</sup>
2,4,6-Trinitrotoluene	7,170.00	MAX			ug/m <sup>3</sup>
2,4-Dinitrotoluene	195.00	MAX			ug/m <sup>3</sup>
2,6-Dinitrotoluene	113.00	MAX			ug/m <sup>3</sup>
2-Amino-4,6-Dinitrotoluene	32.80	MAX			ug/m <sup>3</sup>
4-Amino-2,6-Dinitrotoluene	65.30	MAX			ug/m <sup>3</sup>
Acetaldehyde	1,170.00		91.00		ug/m <sup>3</sup>
Acetone	322.00		206.00		ppbv
Acetonitrile	2,288.48	J	662.45	J	ppbv
Ammonia	10,300,000.00		23,300,000.00		ug/m <sup>3</sup>
Bromodichloromethane	26.80		5.40	U	ppbv
Butanal	42.90		33.00		ug/m <sup>3</sup>
Carbon Dioxide	0.04		0.02		%
Carbon Monoxide	260.00		109.00		ppmv
Chloroform	49.80		5.40	U	ppbv
Crotonaldehyde	9.74		5.03	J	ug/m <sup>3</sup>
Cyanide	0.02		0.01		ug/m <sup>3</sup>
Cyclohexanol	48.55	J			ppbv
Cyclohexanone	14,300.00	D	540.00		ug/m <sup>3</sup>
Decanal	1,770.00		161.00		ug/m <sup>3</sup>
Dibromochloromethane	12.40		5.40	U	ppbv
Ethanol			36.00	J	
Formaldehyde	10,300.00	D	1,470.00		ug/m <sup>3</sup>
Heptanal	53.50		30.30		ug/m <sup>3</sup>
Hexanal	92.60		39.80	J	ug/m <sup>3</sup>
HMX	7.99	MAX			ug/m <sup>3</sup>
Isopropyl Alcohol	262.82	J		J	ppbv
m-Tolualdehyde	88.40		28.10		ug/m <sup>3</sup>
Nitrous Oxide	16,481.00		9,856.00		ppmv
Nonanal	54.60		28.80		ug/m <sup>3</sup>
NOx	0.50		0.50		ppmv
Octanal	59.30		27.90		ug/m <sup>3</sup>
Oxygen	19.20		20.30		%
Pentanal	4.88	J	0.43	U	ug/m <sup>3</sup>
Propanal	439.00		48.50		ug/m <sup>3</sup>
RDX	174.00	MAX			ug/m <sup>3</sup>
Toluene	2.14		5.40	U	ppbv
Total Hydrocarbons	43.60		50.50		ppmv

J = Estimated Value; concentration is below limit of quantification

MAX = Reported result was from a multi-fraction gas sampling train that contains both non-detected results and positive results

U = Analyte was not detected

D = Result obtained from analysis of a dilution or surrogate diluted below detection limit

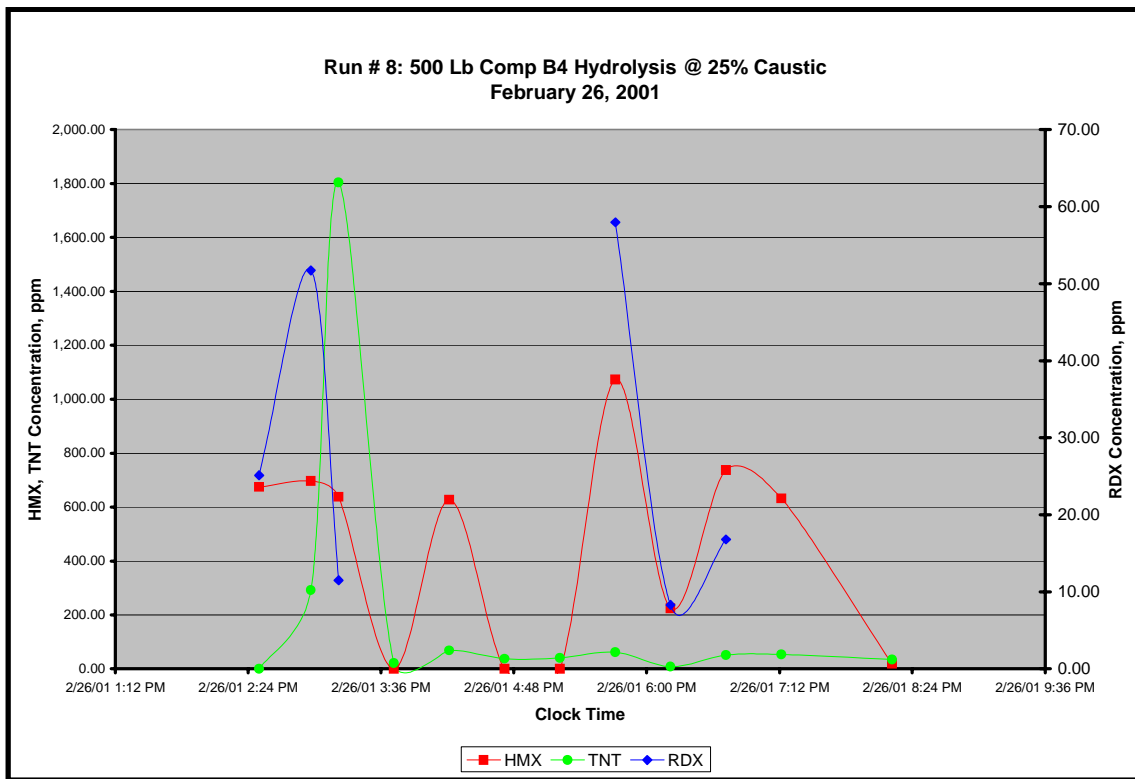


Figure 6-7. Run 8, Mid-Run Destruction Efficiency

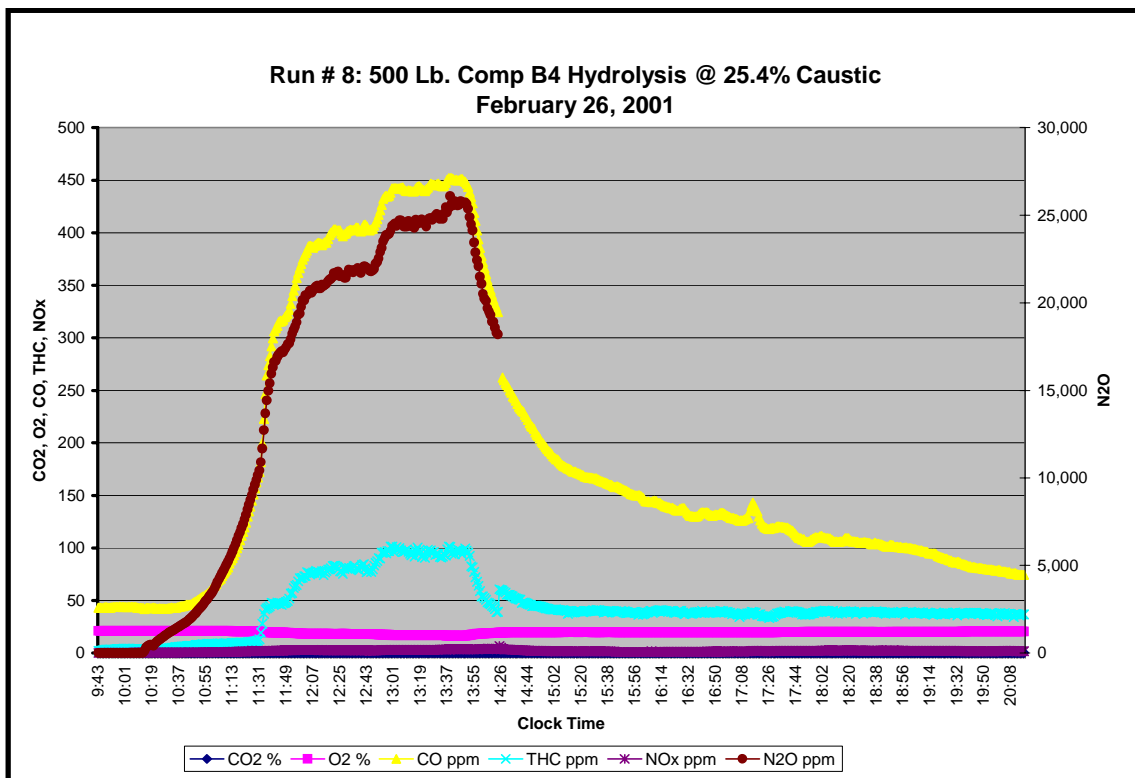


Figure 6-8. Run 8, Off-gas Production



Table 6-6. Run 8, Composition B4 End of Run Hydrolysate Characterization

End of Run Hydrolysate Analysis				
Component	Concentration	Unit	ppm	Note
Acetate	1,110.00	mg/l	1,110.00	
Aluminum	3,770.00	ug/l	3.77	
Ammonia	1,320.00	mg/l	1,320.00	
Beryllium	2.60	ug/l	0.0026	J
Calcium	72,600.00	ug/l	72.6	
Chromium	310.00	ug/l	0.31	J
Cobalt	120.00	ug/l	0.12	J
Copper	1,840.00	ug/l	1.84	
Cyanide (Sodium Cyanide)	136,000.00	ug/l	136	
Fluoride	98.00	mg/l	98.00	J
Formate	12,100.00	mg/l	12,100.00	
Iron	5,900.00	ug/l	5.9	J
Lead	370.00	ug/l	0.37	J
Magnesium	6,660.00	ug/l	6.66	
Manganese	62.00	ug/l	0.062	J
Molybdenum	160.00	ug/l	0.16	J
Nickel	600.00	ug/l	0.6	J
Nitrate-N	10.00	mg/l	10.00	J
Nitrite-N	4,200.00	mg/l	4,200.00	
Potassium	128,000.00	ug/l	128	
Sodium	124,000,000.00	ug/l	124000	
Sulfate	190.00	mg/l	190.00	
Zinc	28,000.00	ug/l	28	
TIC	1,018.50	mg/l	1,018.50	
TOC	3.48	mg/l	3.48	
COD	29,400.00	mg/l	29,400.00	
Total Suspended Solids	817.00	mg/l	817.00	
Total Dissolved Solids	292,000.00	mg/l	292,000.00	
Normality as NaOH	5.15	n		
Density	1.22	g/ml		

J = Estimated Value; concentration is below limit of quantification

Table 6-7. Run 8, Composition B4 Off-gas Characterization

Reactor Off Gas Analysis					
Component	During Energetic Addition	Note	During Reaction	Note	Unit
1,3,5-Trinitrobenzene	7.10	MAX			ug/m <sup>3</sup>
1,3-Dinitrobenzene	15.90	MAX			ug/m <sup>3</sup>
2,4,6-Trinitrotoluene	53.00	MAX			ug/m <sup>3</sup>
2,4-Dinitrotoluene	9,870.00	MAX			ug/m <sup>3</sup>
4-Amino-2,6-Dinitrotoluene	1.51	MAX			ug/m <sup>3</sup>
Acetaldehyde	1,820.00	D	216.00		ug/m <sup>3</sup>
Acetone	436.00		4.40	U	ppbv
Ammonia	18,300,000.00		12,900,000.00		ug/m <sup>3</sup>
Bromodichloromethane	13.40		4.40	U	ppbv
Butanal	62.40		21.00		ug/m <sup>3</sup>
Carbon Dioxide	0.03		0.04		%
Carbon Monoxide	229.00		91.00		ppmv
Chloroform	25.60		4.40	U	ppbv
Crotonaldehyde	0.37	U	6.08		ug/m <sup>3</sup>
Cyanide	0.02		0.01		ug/m <sup>3</sup>
Cyclohexane	394.23	J			ppbv
Cyclohexanone	31,200.00	D	859.00		ug/m <sup>3</sup>
Decanal	6,770.00	D	151.00		ug/m <sup>3</sup>
Formaldehyde	13,100.00	D	1,730.00	B	ug/m <sup>3</sup>
Heptanal	34.50		22.00		ug/m <sup>3</sup>
Hexanal	426.00		63.60		ug/m <sup>3</sup>
Methyl Chloride	186.00	B	143.00	B	ppbv
m-Tolualdehyde	65.30		17.40		ug/m <sup>3</sup>
Nitrous Oxide	14,417.00				ppmv
Nonanal	44.40		1.44	U	ug/m <sup>3</sup>
NOx	2.10		2.00		ppmv
Octanal	46.70		0.58	U	ug/m <sup>3</sup>
Oxygen	19.30		20.10		%
RDX	4.01	MAX			ug/m <sup>3</sup>
Toluene	14.00		4.40	U	ppbv
Total Hydrocarbons	47.10		38.00		ppmv

J = Estimated Value; concentration is below limit of quantification

MAX = Reported result was from a multi-fraction gas sampling train that contains both non-detected results and positive results

U = Analyte was not detected

D = Result obtained from analysis of a dilution or surrogate diluted below detection limit

B = When applied to anions or organic analysis the qualifier indicates that the analyte was detected in the associated method/instrument blank

#### 6.1.2 Discussion & Analysis:

1. The end of run liquid analyses indicates that the destruction rate efficiency achieved approximately 5 hours after cessation of the feeding of Composition B4 explosive to the reactor – 9 hours after the start of the run ranged from 99.9884% to 100.00%. 100% of the HMX and RDX was destroyed, while the TNT destruction rate efficiency ranged from 99.9711 to 100.00%
2. The variability in the energetics concentration in the mid run samples for the Composition B4 runs is most likely due to the fact that some energetic particles are entering and passing through the recycle loop where the samples are being collected by the auto-sampler system.
3. The only “bad actor” detected in the hydrolysate is cyanide (possibly sodium cyanide) at a range of 39 to 139 ppm, increasing in concentration with increasing caustic strength. However, previous work performed under the AWCA program had reported that the hydrolysis of energetics produced cyanide and that the subsequent treatment of said hydrolysate using SCOW technology adequately reduces the hazardous compound concentrations in the hydrolysate feeds; i.e., cyanide was reduced to less than 36 mg/L, well below levels of concern. (PM ACWA, 1999 Supplemental Report to Congress, p. B.4-64 and PM ACWA, 2001 Supplement report to Congress, p C.4-44)) Therefore, at the levels present, no problems are anticipated completing the processing of the hydrolysate using the SCWO system or bioreactor systems.
4. The energetics loading for the three runs with Composition B4 is 6-7 weight-percent resulting in a total solids (dissolved and suspended) loading at the end of the run of between 14-to-29 weight-percent depending on the caustic strength.
5. The airflow across of the reactor headspace was maintained at ~40 scfm during the test. The off gassing (see Figures 6-4, 6-6 and 6-8) tracks nicely with the addition of the Composition B4 explosive and fell off very quickly after the addition was completed, indicating that the most of the reactions are taking place during energetic addition and progressing to completion in line with the liquid analysis data.
6. The heat released by the exothermic reaction easily controlled by the reactor jacket cooling system, and the hydrolysate was maintained at the 87°C set point without difficulty.
7. The operating level in the reactor was maintained just above the lower impeller representing a starting volume of ~700-gallons. At this level, foaming was not a problem since the agitator formed a clearly defined vortex that quickly dispersed the foam back into the solution. The agitator speed was controlled at approximately 70 rpm throughout the test.
8. Examination of the off-gas characterization for the three runs indicates that some energetic materials (1,3,5-Trinitrobenzene, Dinitrotoluene, TNT, RDX, HMX) at low levels were entrained in the gas stream during the addition phase of the process. TNT has a measurable vapor pressure at ambient, therefore one would expect TNT to come-off as part of the off-gassing stream. The presence of RDX and HMX in of the off-gas stream is most likely attributed to these materials becoming entrained in water droplets and evolved with the off gas. Also, some dusting occurs as the Composition B4 falls into the reactor through the headspace air stream, possibly contributing to these readings. The energetic materials disappeared from the air stream once the additional was stopped. Use of a condenser, just above the reactor, to drop any entrained materials back into the reactor should be effective.

9. The only significant “bad actors” identified in the off-gas characterization were cyanide, toluene and benzene; however the levels were extremely low and the subsequent SCOW process will complete the destruction of these compounds.
10. The major constituents of the off gassing were ammonia, nitrous oxide, cyclohexanone and formaldehyde. These gasses can be effectively treated with a scrubber system, with the water from the scrubber then processed through the SCWO system as the final treatment step before release. The average range concentration of CO<sub>2</sub>, O<sub>2</sub>, CO, THC, NO<sub>x</sub> and N<sub>2</sub>O in the off gas stream during energetic addition for the three runs was: 0.03 - .08%, 19.0 – 19.3%, 229 – 284 ppmvd, 27.6 – 47.1 ppmvd, 0.5 – 2.1 ppmvd, 14,005 – 16,481 ppmvd, respectively and during digestion the concentration was: 0.02 - 0.05%, 20.1 – 20.3%, 91 – 112 ppmvd, 38 – 70.8 ppmvd, 0.0 – 2 ppmvd, 9,231 – 9,856 ppmvd, respectively.
11. The inorganic materials (metals) detected in the hydrolysate end of runs analysis are sourced from the sodium hydroxide stock feed that contains some of these components.

## 6.2 M1 Propellant Hydrolysis Tests & Results (Runs 9, 10, 11 and 14).

The objective of these tests is to clearly determine and define the optimum operating parameters for the M1 propellant hydrolysis process to support the design and installation of the full-scale hydrolysis process at the Pueblo Chemical Agent Disposal Facility for the destruction of propellants contained in the 105mm Projectile, Chemical Agent HD, M60.

The table below identifies the process operating parameters for Runs 9, 10, 11, and 14.

Table 6-8. M1 Propellant Test Parameters

Operating Condition	Run 9	Run 10	Run 11	Run 14 Extended
M1 Feed Rate, lbs/hr				
1 <sup>st</sup> hour	50	50	50	50
2 <sup>nd</sup> hour	100	100	100	100
3 <sup>rd</sup> hour	150	150	150	150
4th hour	200	200	200	200
Caustic Soda Concentration, wt%	11.3	20	25	20
Caustic Soda Feed, gal	700	700	700	700
Reactor Operating Temperature, °C	87	87	87	87
Agitation Speed, RPM	70	70	70	70
Date Conducted	2/28/2001	3/2/2001	3/7/2001	3/15/2001

Run 14 was conducted over a 24-hour period to determine if the off gassing would finally cease and an endpoint reached. Off gassing was still taking place after 24-hours had elapsed.

The following charts present the process operating and control parameters for hydrolysis of M1 propellant:

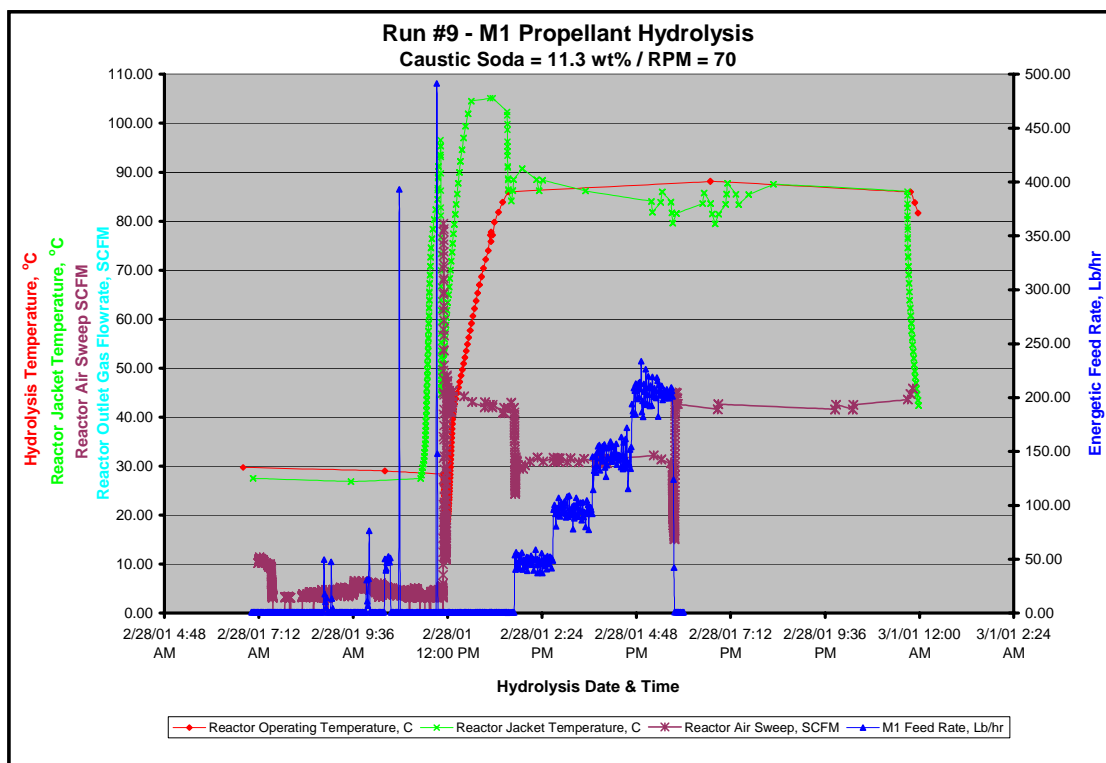


Figure 6-9. Run 9, Process Operating and Control Parameters

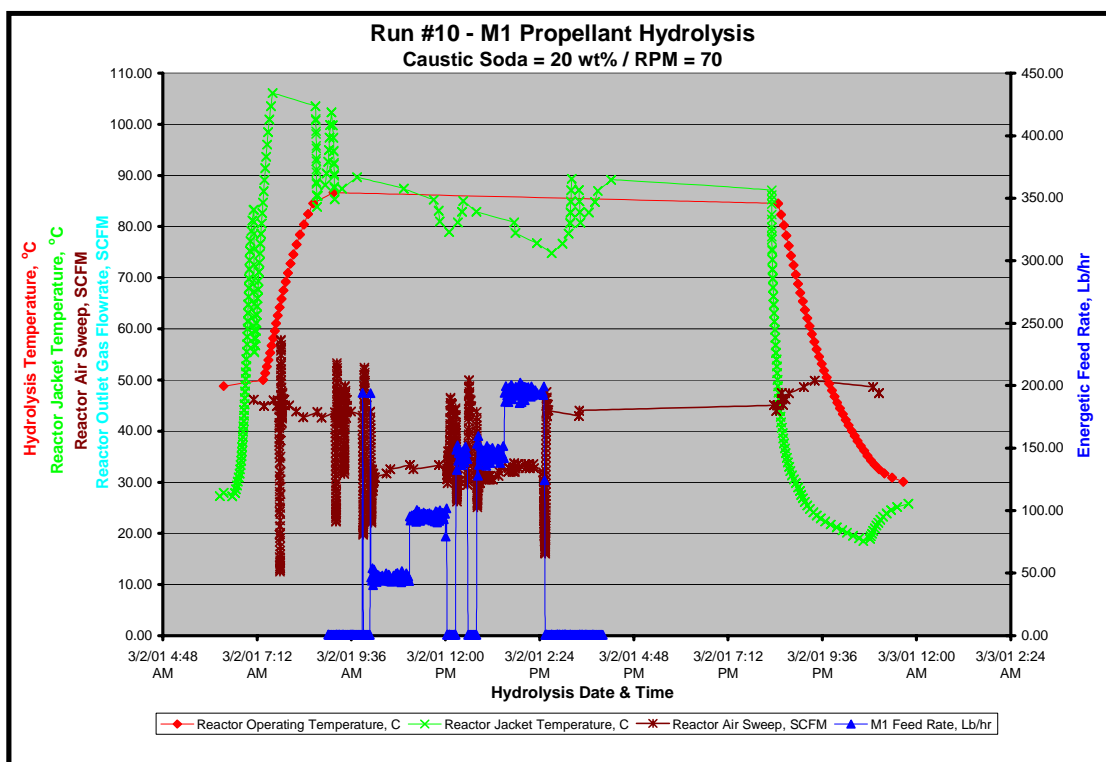


Figure 6-10. Run 10, Process Operating and Control Parameters

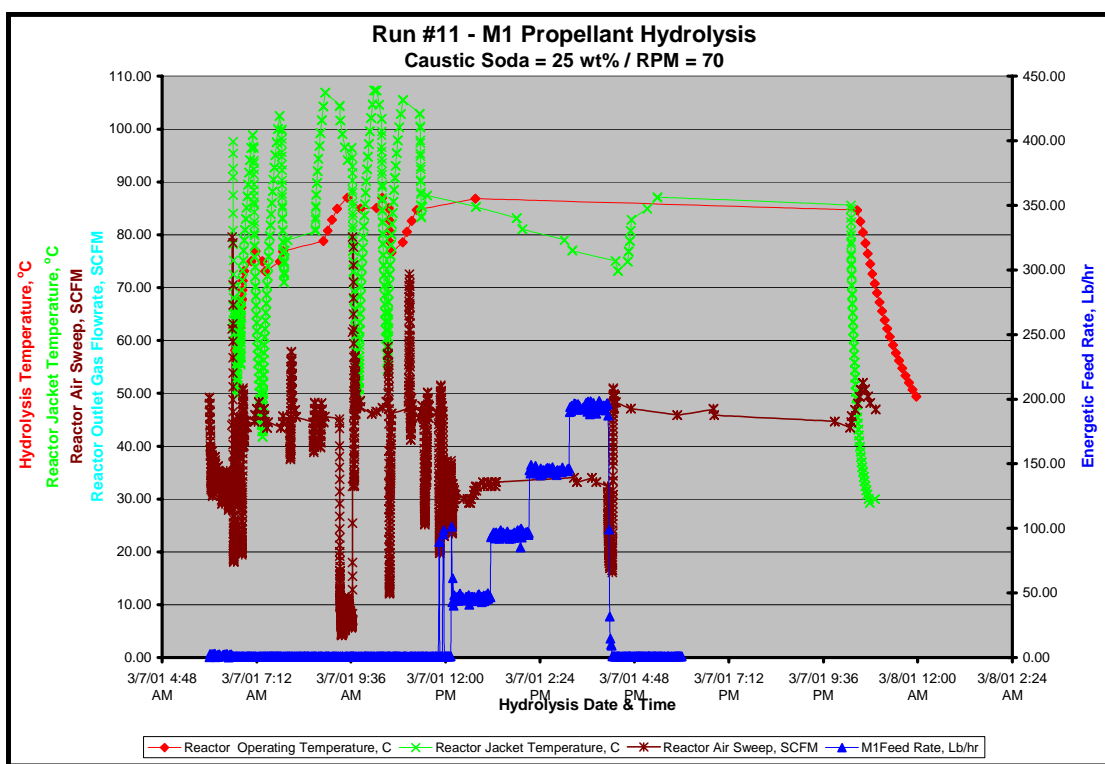


Figure 6-11. Run 11, Process Operating and Control Parameters

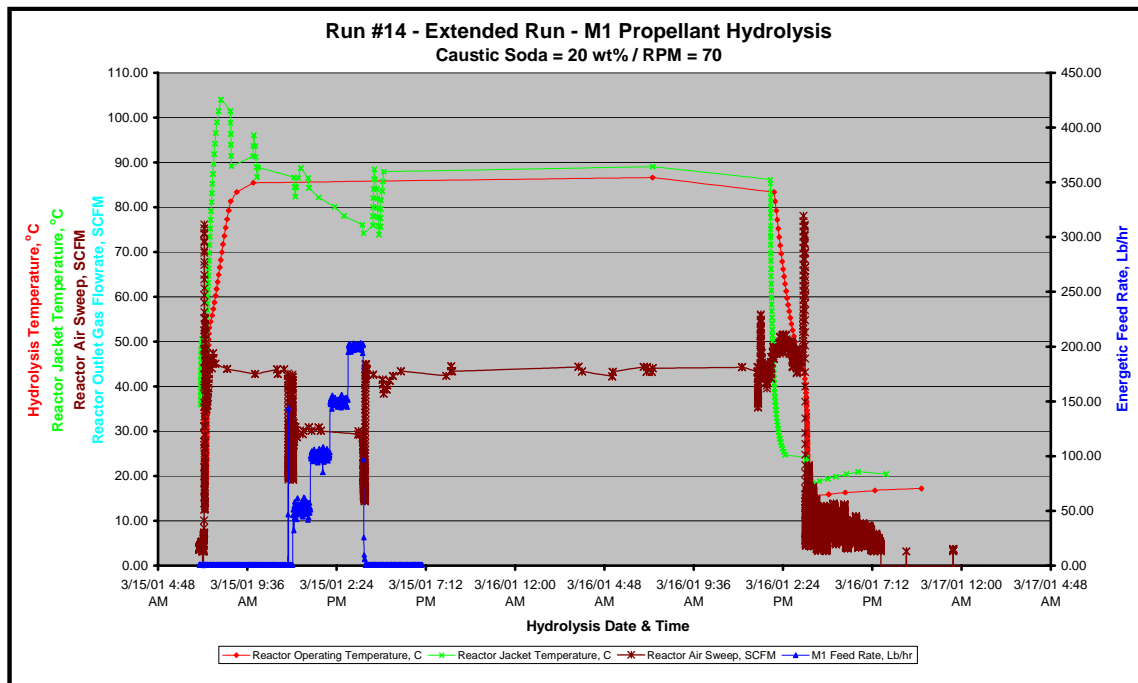


Figure 6-12. Run 14, Process Operating and Control Parameters (Extended Run)

### 6.2.1 Tests Results:

The plots below represent M1 propellant destruction as a function of reactor residence time. Run 14, the extended run was conducted to determine at what time the total hydrocarbon in the off gas stream would start to level off and decrease.

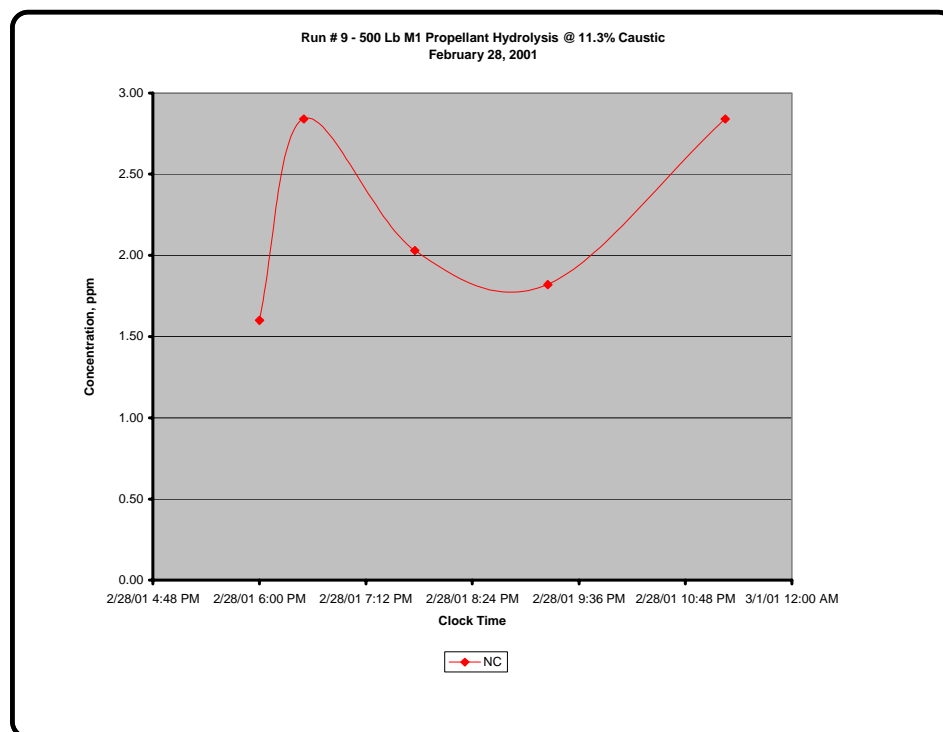


Figure 6-13. Run 9, Mid-Run Destruction Efficiency

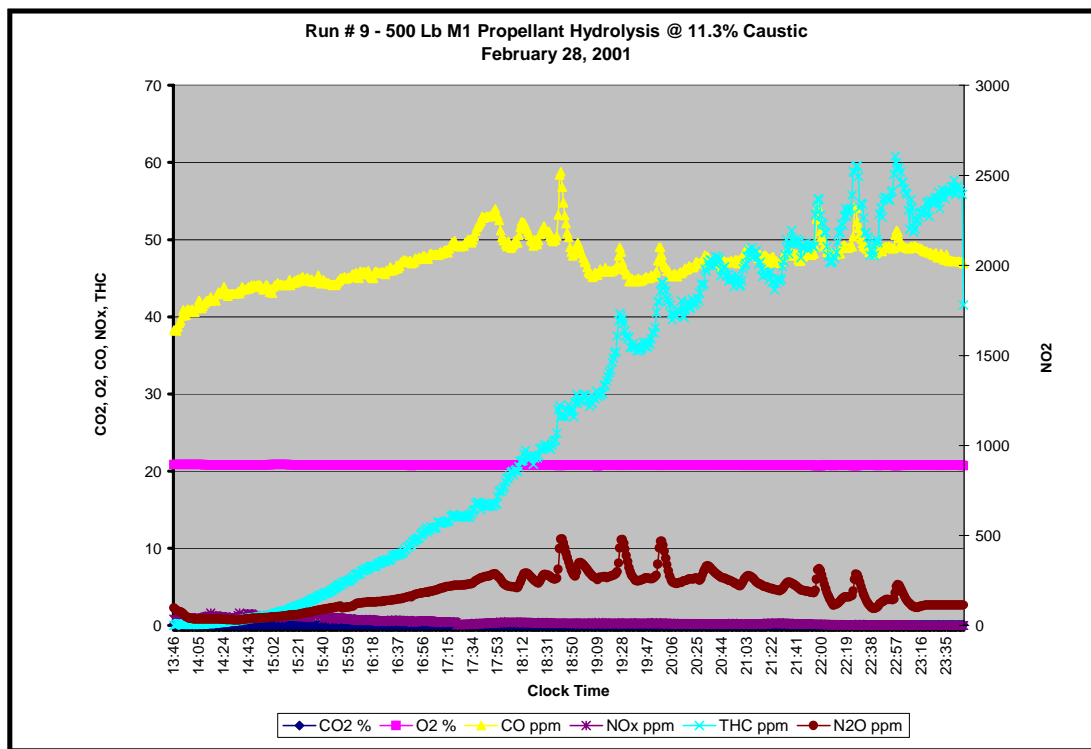


Figure 6-14. Run 9, Off-gas Production



Table 6-9. Run 9, M1 Propellant End of Run Hydrolysate Characterization

End of Run Hydrolysate Analysis				
Component	Concentration	Unit	ppm	Note
2,4-Dinitrotoluene	740.00	ug/l	0.74	J
2-Methylphenol	640.00	ug/l	0.64	J
4-Nitrophenol	1,600.00	ug/l	1.60	J
Acetate	5,120.00	mg/l	5,120.00	
Ammonia	54.00	mg/l	54.00	
Barium	97.50	ug/l	0.10	
bis(2-Ethylhexyl)phthalate	400.00	ug/l	0.40	J
Calcium	40,600.00	ug/l	40.60	
Chloride	40.00	mg/l	40.00	J
Chromium	109.00	ug/l	0.11	
Copper	1,120.00	ug/l	1.12	
Cyanide (Sodium Cyanide)	29,400.00	ug/l	29.40	
Fluoride	589.00	mg/l	589.00	
Formate	1,290.00	mg/l	1,290.00	
Iron	4,000.00	ug/l	4.00	
Magnesium	9,150.00	ug/l	9.15	
Manganese	34.30	ug/l	0.03	
Nickel	104.00	ug/l	0.10	
Nitrate-N	1,480.00	mg/l	1,480.00	
Nitrite-N	4,180.00	mg/l	4,180.00	
Nitrobenzene	260.00	ug/l	0.26	J
Potassium	11,600.00	ug/l	11.60	
Sodium	45,900,000.00	ug/l	45,900.00	
Sulfate	138.00	mg/l	138.00	
Zinc	269.00	ug/l	0.27	
TIC	765.00	mg/l	765.00	
TOC	14,475.00	mg/l	14,475.00	
COD	40,700.00	mg/l	40,700.00	
Total Suspended Solids	640.00	mg/l	640.00	
Total Dissolved Solids	126,000.00	mg/l	126,000.00	
Normality as NaOH	1.30	n		
Density	1.09	g/ml		

J = Estimated Value; concentration is below limit of quantification

Table 6-10. Run 9, M1 Propellant Off-gas Characterization

Reactor Off Gas Analysis					
Component	During Energetic Addition	Note	During Reaction	Note	Unit
2-Butanone	570.00	U	1,040.00		ppbv
Acetaldehyde	15,200.00	D	15,700.00	D	ug/m <sup>3</sup>
Acetone	29,900.00		78,200.00		ppbv
Ammonia	198,000.00		618,000.00		ug/m <sup>3</sup>
Benzene	15.00	U	43.20		ppbv
Bromodichloromethane	26.40		14.00	U	ppbv
Butanal	92.80		389.00		ug/m <sup>3</sup>
Carbon Dioxide	0.04		0.04		%
Carbon Monoxide	2.00		5.00		ppmv
Chloroform	24.20		14.00	U	ppbv
Cyanide	0.01	U	0.02		mg/m <sup>3</sup>
Cyclohexane	133,053.00	J	248,135.40	J	ppbv
Cyclohexanone	1,110.00		120.00		ug/m <sup>3</sup>
Decanal	162.00		22.90		ug/m <sup>3</sup>
Dibromochloromethane	21.90		14.00	U	ppbv
Ethyl ether	17,673.00	J	36,905.00	J	ppbv
Formaldehyde	688.00	B	251.00	B	ug/m <sup>3</sup>
Heptanal	252.00		53.30		ug/m <sup>3</sup>
Hexanal	165.00		44.20		ug/m <sup>3</sup>
Methyl Chloride	31.60	B	26.40	B	ppbv
m-Tolualdehyde	48.10		40.10		ug/m <sup>3</sup>
Nitrous Oxide	113.00		232.00		ppmv
Nonanal	22.70		15.20		ug/m <sup>3</sup>
NOx	0.70		0.00		ppmv
Octanal	0.46	U	14.10		ug/m <sup>3</sup>
Oxygen	21.30		21.20		%
Pentanal	27.50		26.40		ug/m <sup>3</sup>
Toluene	12,100.00		42,200.00		ppbv
Xylenes	22.50	U	34.70		ppbv
Total Hydrocarbons	240.30		1,680.70		ppmv

- J = Estimated Value; concentration is below limit of quantification  
 MAX = Reported result was from a multi-fraction gas sampling train that contains both non-detected results and positive results.  
 U = Analyte was not detected  
 D = Result obtained from analysis of a dilution or surrogate diluted below detection limit  
 B = When applied to anions or organic analysis the qualifier indicates that the analyte was detected in the associated method/instrument blank

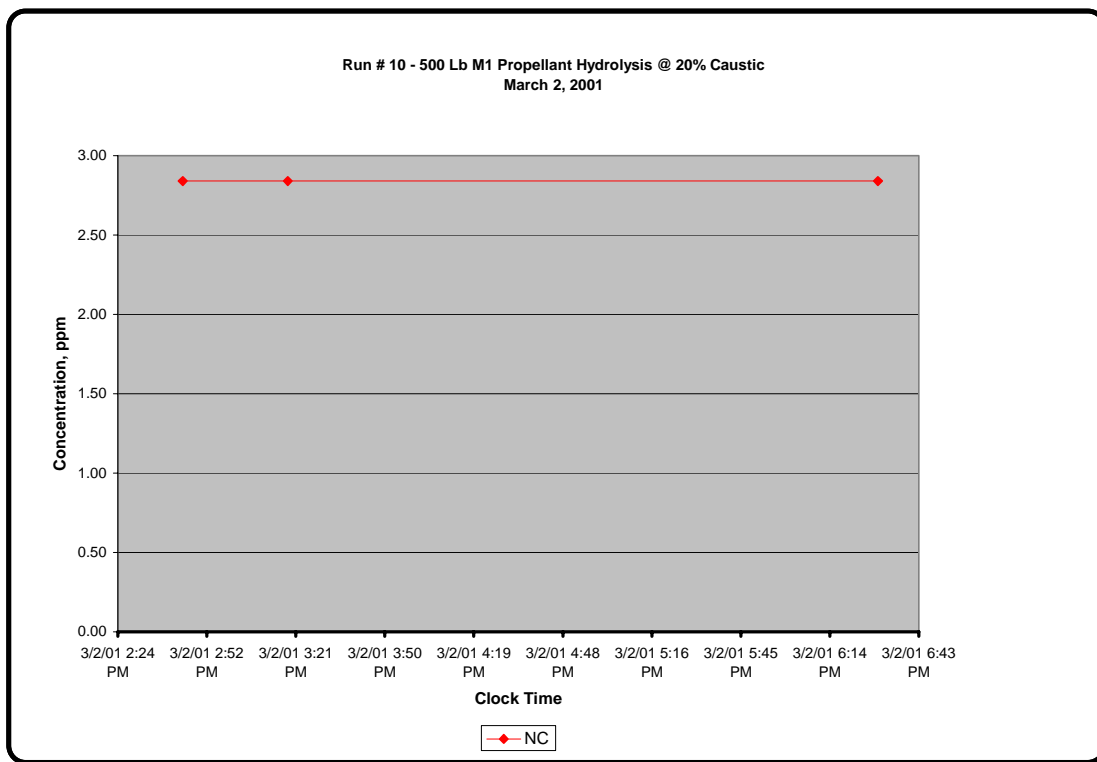


Figure 6-15. Run 10, Mid-Run Destruction Efficiency

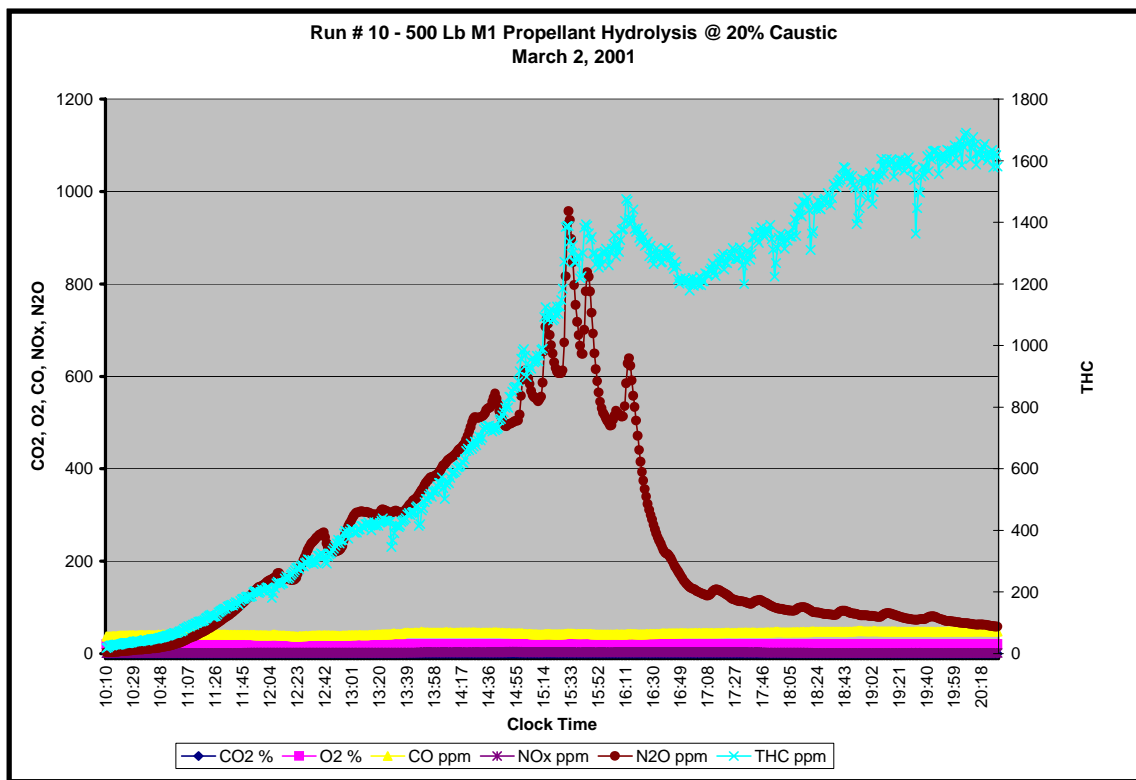


Figure 6-16. Run 10, Off-gas Production

Table 6-11. Run 10, M1 Propellant End of Run Hydrolysate Characterization

End of Run Hydrolysate Analysis				
Component	Concentration	Unit	ppm	Note
2-Methylphenol	5,600.00	ug/l	5.60	J
4-Nitrophenol	3,000.00	ug/l	3.00	
Acetate	6,130.00	mg/l	6,130.00	
Aluminum	393.00	ug/l	0.39	
Ammonia	55.50	mg/l	55.50	
Barium	174.00	ug/l	0.17	
Calcium	34,500.00	ug/l	34.50	
Chromium	176.00	ug/l	0.18	
Cobalt	104.00	ug/l	0.10	
Copper	941.00	ug/l	0.94	
Cyanide (Sodium Cyanide)	76,000.00	ug/l	76.00	J
Di-n-octylphthalate	3,000.00	ug/l	3.00	
Fluoride	851.00	mg/l	851.00	
Formate	1,740.00	mg/l	1,740.00	
Iron	6,170.00	ug/l	6.17	
Magnesium	7,490.00	ug/l	7.49	J
Manganese	45.90	ug/l	0.05	
Molybdenum	28.80	ug/l	0.03	
Nickel	176.00	ug/l	0.18	
Nitrate-N	1,780.00	mg/l	1,780.00	
Nitrite-N	5,060.00	mg/l	5,060.00	J
o-Phosphate-P	526.00	mg/l	526.00	
Phosphorus	122.00	ug/l	0.12	
Potassium	43,900.00	ug/l	43.90	
Sodium	92,840,000.00	ug/l	92,840.00	
Sulfate	196.00	mg/l	196.00	
Zinc	435.00	ug/l	0.44	
TIC	1,121.75	mg/l	1,121.75	
TOC	17,825.00	mg/l	17,825.00	
COD	19,600.00	mg/l	19,600.00	
Total Suspended Solids	1,280.00	mg/l	1,280.00	
Total Dissolved Solids	243,000.00	mg/l	243,000.00	
Normality as NaOH	3.75	n		
Density	1.18	g/ml		

J = Estimated Value; concentration is below limit of quantification

Table 6-12. Run 10, M1 Propellant Off-gas Characterization

Reactor Off Gas Analysis					
Component	During Energetic Addition	Note	During Reaction	Note	Unit
1,3,5-Trinitrobenzene	8.41	MAX			ug/m <sup>3</sup>
1-Butanol	161,586.80	J			ppbv
2,4,6-Trinitrotoluene	119.00	MAX			ug/m <sup>3</sup>
2,4-Dinitrotoluene	716.00				ug/m <sup>3</sup>
2,6-Dinitrotoluene	3.37	MAX			ug/m <sup>3</sup>
2-Amino-4,6-Dinitrotoluene	3.13	MAX			ug/m <sup>3</sup>
4-Amino-2,6-Dinitrotoluene	3.34	MAX			ug/m <sup>3</sup>
Acetaldehyde	16,300.00	D	11,600.00	D	ug/m <sup>3</sup>
Acetone	37,800.00		89,000.00		ppbv
Ammonia	131,000.00		616,000.00		ug/m <sup>3</sup>
Butanal	123.00		807.00		ug/m <sup>3</sup>
Carbon Dioxide	0.03		0.03		%
Carbon Monoxide	0.00		3.00		ppmv
Crotonaldehyde	0.33	U	9.03		ug/m <sup>3</sup>
Cyanide	0.01	U	0.01		mg/m <sup>3</sup>
Cyclohexanone	49.10		68.10		ug/m <sup>3</sup>
Decanal	19.30		12.50		ug/m <sup>3</sup>
Ethyl ether	58,147.10	J	138,181.80	J	ppbv
Formaldehyde	300.00	B	128.00	B	ug/m <sup>3</sup>
Heptanal	23.60		24.40		ug/m <sup>3</sup>
Hexanal	124.00		64.30		ug/m <sup>3</sup>
HMX	4.31	MAX			ug/m <sup>3</sup>
Methyl Chloride	753.00	B	1,620.00	B	ppbv
m-Tolualdehyde	25.30		35.60		ug/m <sup>3</sup>
Nitrous Oxide	201.00		257.00		ppmv
Nonanal	16.80		13.90		ug/m <sup>3</sup>
NOx	0.60		1.00		ppmv
Octanal	15.80		12.90		ug/m <sup>3</sup>
Oxygen	21.30		21.30		%
Pentanal	35.60		15.30		ug/m <sup>3</sup>
RDX	9.77				ug/m <sup>3</sup>
Toluene	15,400.00		30,900.00		ppbv
Total Hydrocarbons	286.00		1,336.00		ppmv

J = Estimated Value; concentration is below limit of quantification

MAX = Reported result was from a multi-fraction gas sampling train that contains both non-detected results and positive results.

U = Analyte was not detected

D = Result obtained from analysis of a dilution or surrogate diluted below detection limit

B = When applied to anions or organic analysis the qualifier indicates that the analyte was detected in the associated method/instrument blank

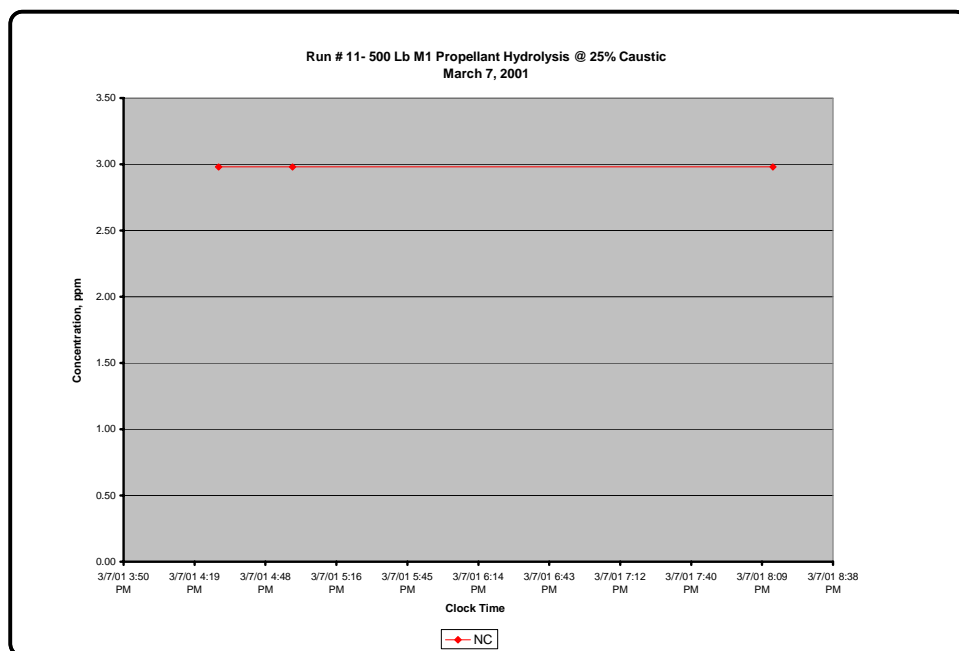


Figure 6-17. Run 11, Mid-Run Destruction Efficiency

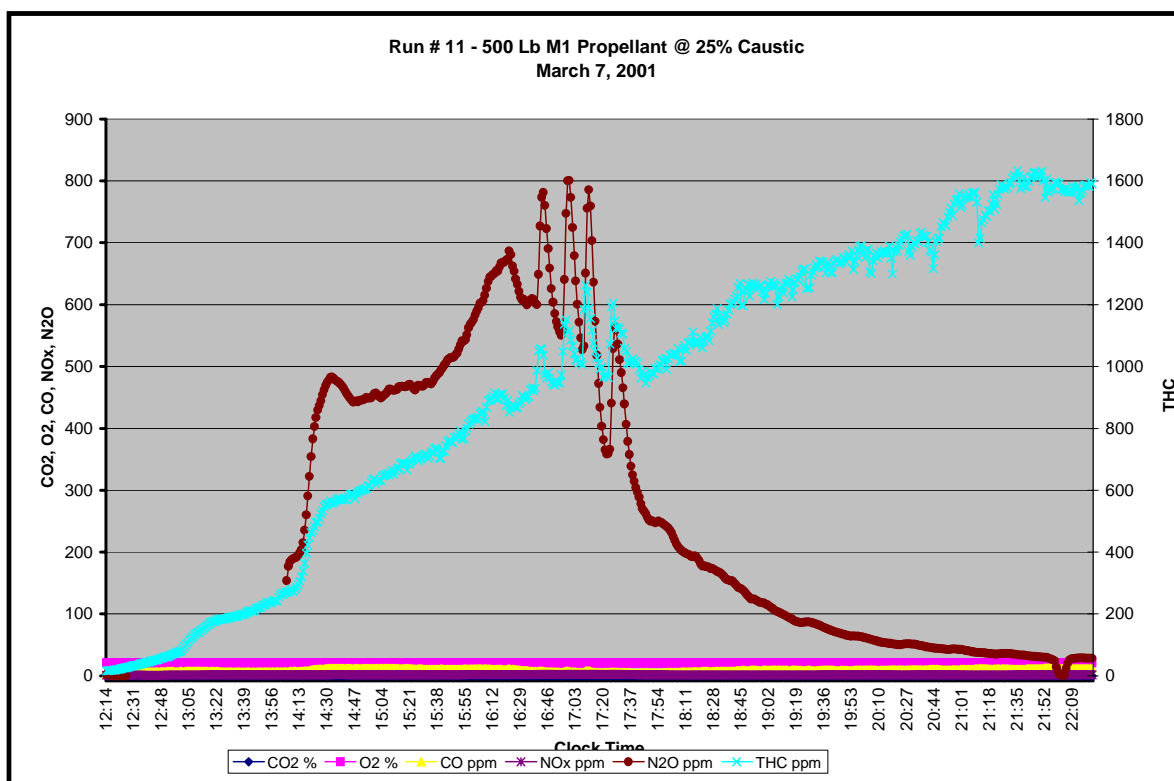


Figure 6-18. Run 11, Off-gas Production

Table 6-13. Run 11, M1 Propellant End of Run Hydrolysate Characterization

End of Run Hydrolysate Analysis				
Component	Concentration	Unit	ppm	Note
2-Methylphenol	1,400.00	ug/l	1.40	J
4-Nitrophenol	1,500.00	ug/l	1.50	J
Acetate	7,350.00	mg/l	7,350.00	
Aluminum	418.00	ug/l	0.42	
Ammonia	55.50	mg/l	55.50	
Barium	166.00	ug/l	0.17	
Calcium	30,000.00	ug/l	30.00	
Chloride	8,900.00	mg/l	8,900.00	
Chromium	221.00	ug/l	0.22	
Cobalt	101.00	ug/l	0.10	
Copper	843.00	ug/l	0.84	
Cyanide (Sodium Cyanide)	95,400.00	ug/l	95.40	
Fluoride	10,200.00	mg/l	10,200.00	
Formate	2,240.00	mg/l	2,240.00	
Iron	6,750.00	ug/l	6.75	
Magnesium	6,580.00	ug/l	6.58	
Manganese	59.20	ug/l	0.06	
Molybdenum	28.00	ug/l	0.03	J
Nickel	244.00	ug/l	0.24	
Nitrate-N	845.00	mg/l	845.00	
Nitrite-N	1,860.00	mg/l	1,860.00	
o-Phosphate-P	2,040.00	mg/l	2,040.00	
Phosphorus	498.00	ug/l	0.50	J
Potassium	57,200.00	ug/l	57.20	
Sodium	113,000,000.00	ug/l	113,000.00	
Sulfate	3,280.00	mg/l	3,280.00	
Zinc	442.00	ug/l	0.44	
TIC	1,167.50	mg/l	1,167.50	
TOC	16,825.00	mg/l	16,825.00	
COD	49,600.00	mg/l	49,600.00	
Total Suspended Solids	600.00	mg/l	600.00	
Total Dissolved Solids	295,000.00	mg/l	295,000.00	
Normality as NaOH	4.95	n		
Density	1.22	g/ml		

J = Estimated Value; concentration is below limit of quantification

Table 6-14. Run 11, M1 Propellant Off-gas Characterization

Reactor Off Gas Analysis					
Component	During Energetic Addition	Note	During Reaction	Note	Unit
1,3,5-Trinitrobenzene	2.21	MAX			ug/m <sup>3</sup>
1-Butanol	14,568.70	J	49,442.90	J	ppbv
2,4,6-Trinitrotoluene	43.90	MAX			ug/m <sup>3</sup>
2,4-Dinitrotoluene	21,600.00				ug/m <sup>3</sup>
2,6-Dinitrotoluene	2.97	MAX			ug/m <sup>3</sup>
4-Amino-2,6-Dinitrotoluene	1.63	MAX			ug/m <sup>3</sup>
Acetaldehyde	13,400.00	D	11,200.00	D	ug/m <sup>3</sup>
Acetone	44,800.00		50,800.00		ppbv
Ammonia	81,400.00		544,000.00		ug/m <sup>3</sup>
Butanal	203.00		1,000.00		ug/m <sup>3</sup>
Carbon Dioxide	0.03		0.04		%
Carbon Monoxide	2.00		1.00		ppmv
Crotonaldehyde	8.17		5.71		ug/m <sup>3</sup>
Cyanide	0.01	U	0.01		mg/m <sup>3</sup>
Cyclohexanone	22.70		79.10		ug/m <sup>3</sup>
Decanal	4.05	U	18.40		ug/m <sup>3</sup>
Ethyl ether	88,941.20	J	103,976.50	J	ppbv
Formaldehyde	259.00	B	99.60	B	ug/m <sup>3</sup>
Heptanal	46.70		30.60		ug/m <sup>3</sup>
Hexanal	175.00		60.70		ug/m <sup>3</sup>
Methyl Chloride	906.00	B	598.00	B	ppbv
m-Tolualdehyde	24.90		13.40		ug/m <sup>3</sup>
Nitrous Oxide	465.00		202.00		ppmv
Nonanal	2.29	U	19.80		ug/m <sup>3</sup>
NOx	0.50		0.60		ppmv
Octanal	0.92	U	19.10		ug/m <sup>3</sup>
Oxygen	21.10		21.20		%
Pentanal	86.20		49.00		ug/m <sup>3</sup>
RDX	4.78	MAX			ug/m <sup>3</sup>
Toluene	22,400.00		18,100.00		ppbv
Total Hydrocarbons	400.00		1,248.00		ppmv

J = Estimated Value; concentration is below limit of quantification

MAX = Reported result was from a multi-fraction gas sampling train that contains both non-detected results and positive results.

U = Analyte was not detected

D = Result obtained from analysis of a dilution or surrogate diluted below detection limit

B = When applied to anions or organic analysis the qualifier indicates that the analyte was detected in the associated method/instrument blank



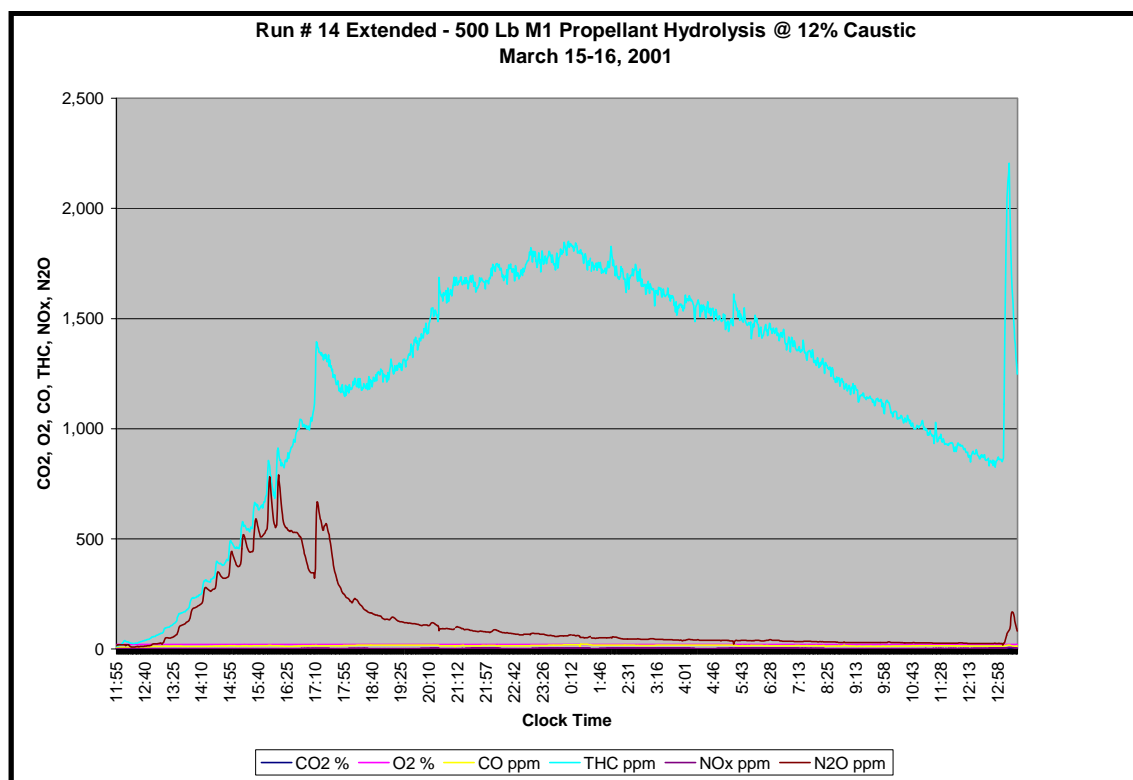


Figure 6-19. Run 14, Off-gas Production

The THC levels did not fall off until nearly 12 hours into the 24-hour run. The spike in the THC level at the ~1300 hour clock time was induced by increasing the agitation speed; thereby supporting the hypothesis that there is significant amounts of dissolved gas in the hydrolysate.

#### 6.2.2 Discussion & Analysis:

1. The end of run liquid analyses indicates that the destruction efficiency achieved approximately 6 hours after cessation of the feeding of M1 propellant to the reactor – 10 hours after the start of the run – ranged from 99.9988% to 100.00%. DNT was detected in the end of run analysis for Run 9.
2. The energetics loading for the four runs with M1 propellant is 6.8 - 8.2 weight-percent resulting in a total solids (dissolved and suspended) loading at the end of the run of between 12.7-to-29.6 weight-percent depending on the caustic strength.
3. The only “bad actor” detected in the hydrolysate is cyanide (possibly sodium cyanide) at a range of 29 to 95 ppm, increasing in concentration with increasing caustic strength. As stated in paragraph 6.1.2 above, the hydrolysis of energetics will produce cyanide and that the subsequent treatment of said hydrolysate using SCOW technology will adequately reduce the hazardous compound concentrations in the hydrolysate feeds well below levels of concern
4. The airflow across of the reactor headspace was maintained at ~40 scfm during the test. The off gassing continued to increase after the addition of the propellant was completed (see Figures 6-14, 6-16, and 6-18), indicating that the reaction was progressing to completion in line with the liquid analysis data. However, the THCs showed no reduction and actually were increasing when the run point was terminated after approximately 10

hours. Run 14, the extended run was conducted to see if an endpoint could be reached regarding off gassing after 24-hours. At 23 hours into the 24-hour run, the agitator speed was increased to see if the off gassing would be effected, and immediately the THC's released increased dramatically (see Figure 6-19). It is conjectured that the off gassing is the results of dissolved gasses and the continued reaction of the caustic solution with the by-products of the destruction of the NC chain (M1 propellant contains 84% NC). Increasing the agitation simply allowed dissolved gasses to be released from the hydrolysate.

5. The heat released by the exothermic reaction easily controlled by the reactor jacket cooling system, and the hydrolysate was maintained at the 87°C set point without difficulty.
6. The operating level in the reactor was maintained just above the lower impeller representing a starting volume of ~700-gallons. Foaming was not experienced with M1 propellant.
7. Examination of the off-gas characterization for the four runs indicates that trace amounts of TNT 1,3,5-trinitrobenzene, Dinitrotoluene (most likely from nitration of DNT used in the M1 propellant at a level of 9%) and HMX and RDX (most likely line residuals from the Composition B and B4 runs) were entrained in the gas stream during the addition phase of the process. All energetics disappeared once the M1 propellant addition was completed. Use of a condenser just above the reactor to drop any entrained materials back into the reactor should be effective.
8. The only significant "bad actors" identified in all four of the off-gas characterizations were toluene, xylene and benzene. These components and other in the off gas stream can be effectively treated with a scrubber system, with the water from the scrubber then processed through the SCWO system as the final treatment step before release.
9. The other major constituents of the off gassing were ammonia, acetone, acetaldehyde, and ethyl ether. These gasses can be effectively treated with a scrubber system, with the water from the scrubber then processed through the SCWO system as the final treatment step before release. The average range concentration of CO<sub>2</sub>, O<sub>2</sub>, CO, THC, NO<sub>x</sub> and N<sub>2</sub>O in the off gas stream during energetic addition for the three runs was: 0.03 - .04%, 21.2 – 21.3%, 0.0 – 2.0 ppmvd, 24.3 – 400.0 ppmvd, 0.5 – 0.7 ppmvd, 113 – 465 ppmvd, respectively and during digestion the concentration was: 0.03 - 0.04%, 21.2 – 21.3%, 1 - 5 ppmvd, 1,248 – 1,680 ppmvd, 0.0 – 1 ppmvd, 202 – 257 ppmvd, respectively.
10. The inorganic materials (metals) detected in the hydrolysate end of runs analysis are sourced from the sodium hydroxide stock feed that contains some of these components.

### 6.3. Unleaded M28 Propellant Hydrolysis Tests & Results (Runs 12 & 13).

The objective of these tests is to clearly determine and define the optimum operating parameters for the M28 propellant hydrolysis process to support the design and installation of the full-scale hydrolysis process at the Pueblo Chemical Agent Disposal Facility for the destruction of propellants contained in the 115mm Rocket, Chemical Agent GB, M56; 115mm Rocket Warhead, Chemical Agent GB, M55; 115mm Rocket, Chemical Agent VX, M55; and 115mm Rocket Warhead, Chemical Agent GB, M55.

The inventory of unleaded M28 granular surrogate propellant (produced at Radford AAP to support the ACWA program) was only sufficient to support two test runs. The M28 surrogate propellant was produced in granulated form as opposed to the cast rocket grain form used in the end item because of cost. Both a leaded version (to the military specification) and an unleaded version (to avoid producing a lead containing waste) of the M28 surrogate propellant were produced at Radford AAP. Figure 6-20 below illustrates the difference between the granulated surrogate propellant used for test purposes and the actual configuration of the cast M28 propellant grains found in the end items. The grains segments pictured were sourced from the propellant surveillance program at Picatinny Arsenal and used to support the grinding study. Consequently, the grains do not have the fluted perforation as the actual propellant grain configuration produced for the end item is shown in Figure 6-21.

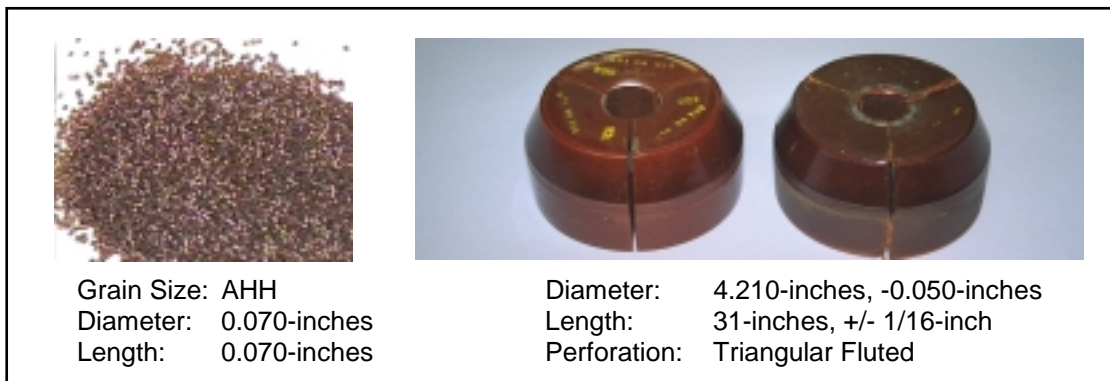


Figure 6-20. M28 Surrogate propellant and Surveillance Propellant Grain Segments

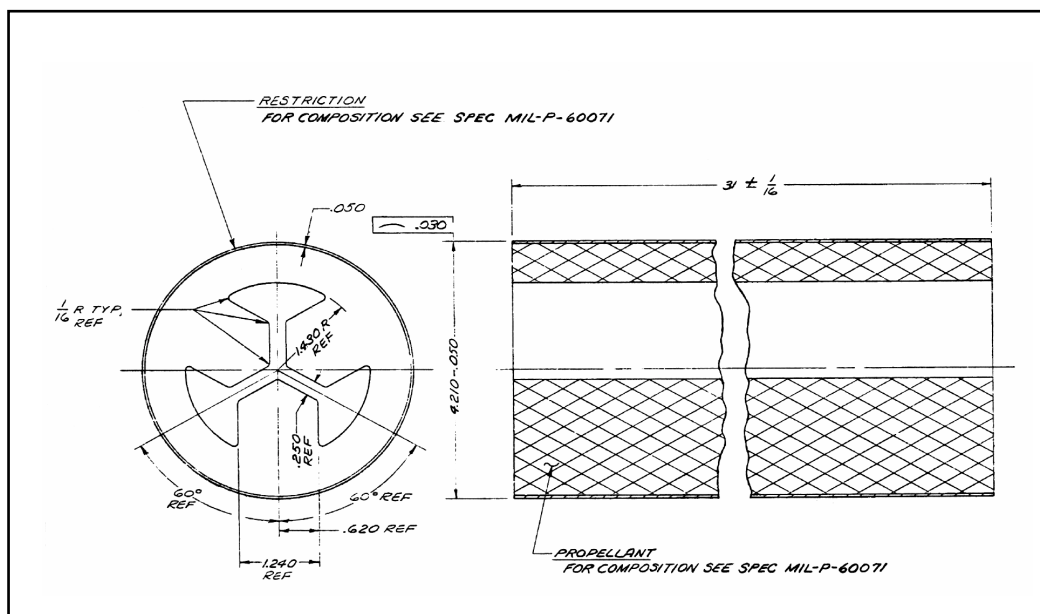


Figure 6-21. M28 Propellant Grain for 115mm Rocket Motor

The table below identifies the process operating and control parameters for Runs 12 and 13.

Table 6-15. M28 Propellant (Unleaded) Test Parameters

Operating Condition	Run 12	Run 13
M28 Feed Rate, lbs/hr		
1 <sup>st</sup> hour	50	50
2 <sup>nd</sup> hour	100	100
3 <sup>rd</sup> hour	150	150
4 <sup>th</sup> hour	200	200
Caustic Soda Concentration, wt%	11.3	20
Caustic Soda Feed, gal	700	700
Reactor Operating Temperature, °C	87	87
Agitation Speed, RPM	70	70
Date Conducted	3/9/2001	3/13/2001

The following charts present the process operating and control parameters for hydrolysis of M28 propellant:

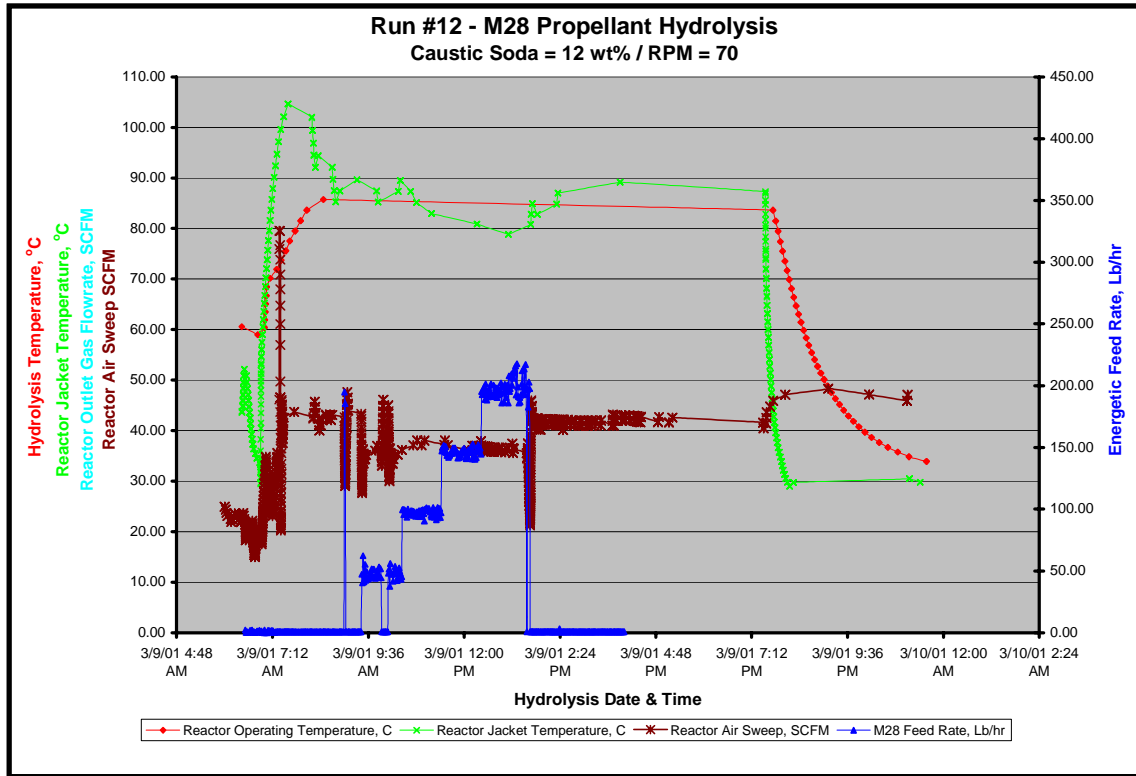


Figure 6-22. Run 12, Process Operating and Control Parameters

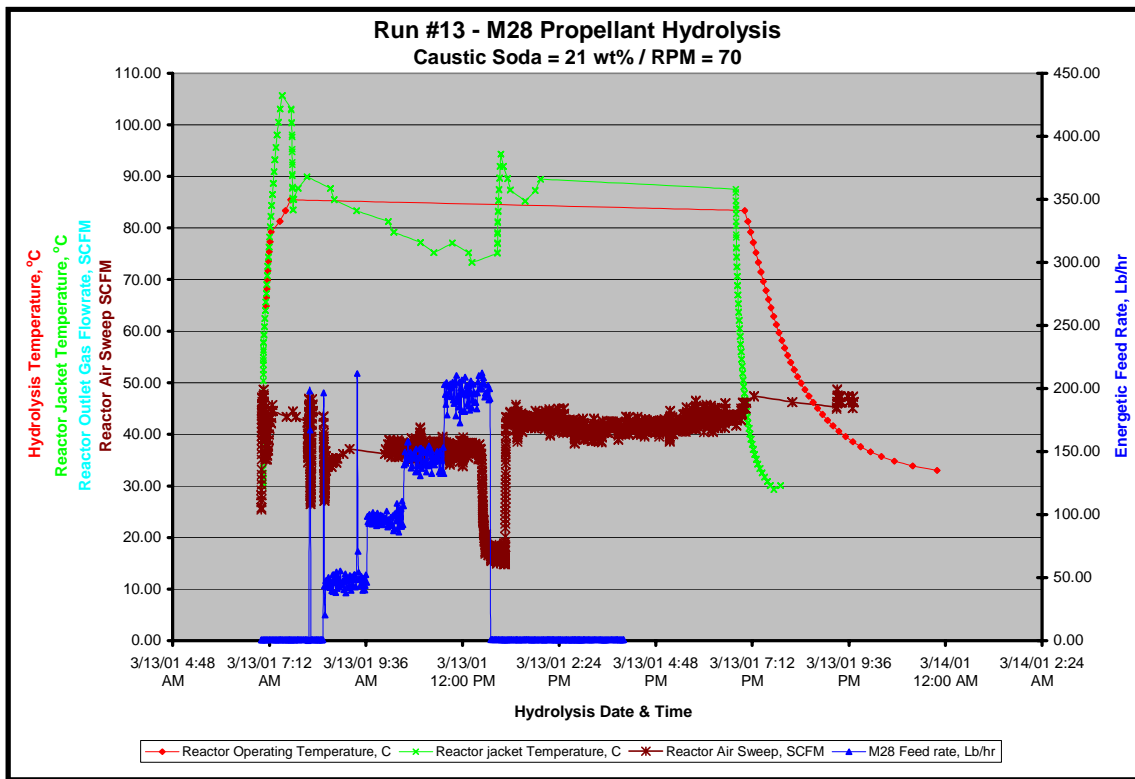


Figure 6-23. Run 13, Process Operating and Control Parameters

### 6.3.1 Tests Results:

The following plots represent the destruction of M28 propellant as a function of reactor residence time.

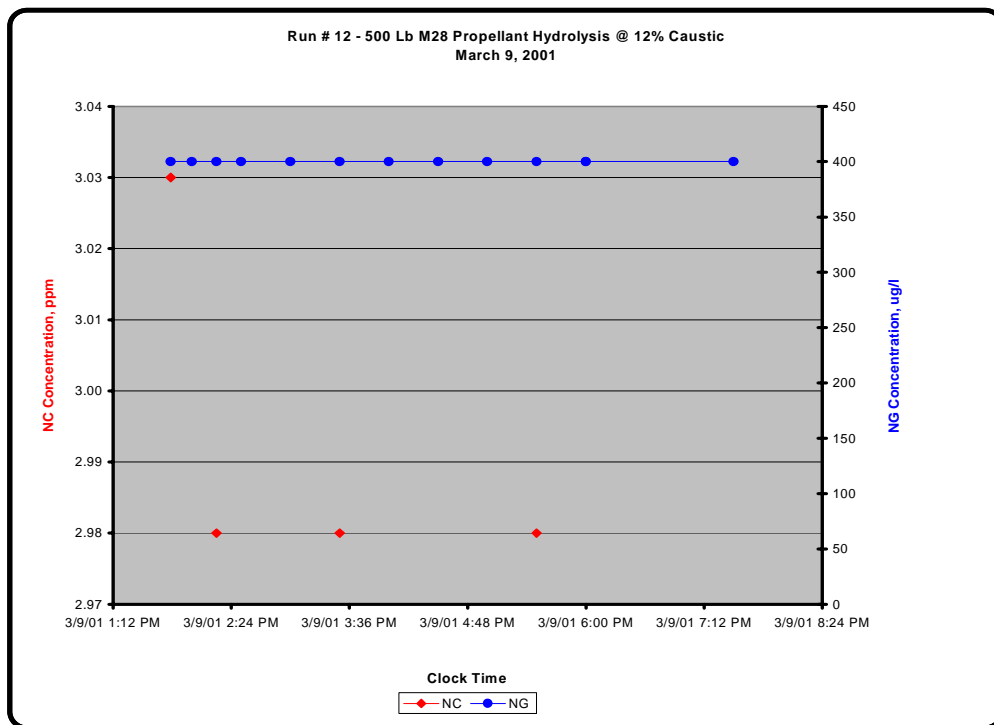


Figure 6-24. Run 12, Mid-Run Destruction Efficiency

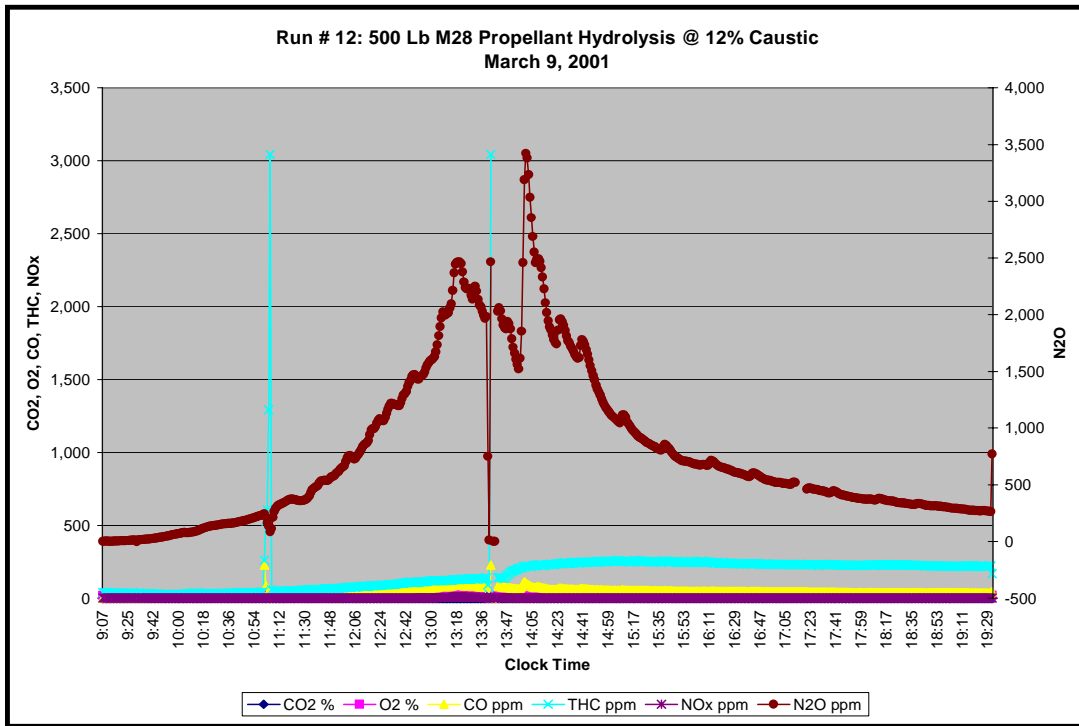


Figure 6-25. Run 12, Off-gas Production

Table 6-16. Run 12, M28 Propellant (Unleaded) End of Run Hydrolysate Characterization

End of Run Hydrolysate Analysis				
Component	Concentration	Unit	ppm	Note
Acetate	14,600.00	mg/l	14,600.00	
Aluminum	487.00	ug/l	0.487	
Ammonia	105.00	mg/l	105.00	
Barium	99.30	ug/l	0.0993	
Calcium	38,200.00	ug/l	38.2	
Chloride	32.30	mg/l	32.30	
Chromium	126.00	ug/l	0.126	
Copper	1,600.00	ug/l	1.6	
Cyanide (Sodium Cyanide)	208,000.00	ug/l	208	
Fluoride	949.00	mg/l	949.00	
Formate	2,360.00	mg/l	2,360.00	
Magnesium	8,000.00	ug/l	8	
Molybdenum	27.80	ug/l	0.0278	J
Nickel	176.00	ug/l	0.176	
Nitrate-N	2,090.00	mg/l	2,090.00	
Nitrite-N	4,760.00	mg/l	4,760.00	
N-Nitrosodiphenylamine	2,700.00	ug/l	2.7	J
Phenol	920.00	ug/l	0.92	J
Phosphorus	490.00	ug/l	0.49	J
Potassium	8,890.00	ug/l	8.89	J
Sodium	50,890,000.00	ug/l	50890	
Sulfate	135.00	mg/l	135.00	
Zinc	268.00	ug/l	0.268	
TIC	1,127.50	mg/l	1,127.50	
TOC	16,375.00	mg/l	16,375.00	
COD	46,300.00	mg/l	46,300.00	
Total Suspended Solids	740.00	mg/l	740.00	
Total Dissolved Solids	153,000.00	mg/l	153,000.00	
Normality as NaOH	1.35	n		
Density	1.10	g/ml		

J = Estimated Value; concentration is below limit of quantification

Table 6-17. Run 12, M28 Propellant (Unleaded) Off-gas Characterization

Reactor Off Gas Analysis					
Component	During Energetic Addition	Note	During Reaction	Note	Unit
1-Butanol	3,456.50	J	725.00	J	ppbv
2-Butanone	722.00	U	4,030.00		ppbv
Acetaldehyde	16,600.00	D	18,400.00	D	ug/m <sup>3</sup>
Acetone	29,100.00		34,000.00		ppbv
Acetonitrile	1,702.30	J			ppbv
Ammonia	159,000.00		1,070,000.00		ug/m <sup>3</sup>
Benzene	184.00		390.00		ppbv
Butanal	325.00		1,160.00		ug/m <sup>3</sup>
Carbon Dioxide	0.04		0.05		%
Carbon Monoxide	34.00		45.00		ppmv
Chloroform	21.60		60.00	U	ppbv
Crotonaldehyde	435.00		8.22		ug/m <sup>3</sup>
Cyanide	0.01		0.08		mg/m <sup>3</sup>
Cyclohexanone	64.70		103.00		ug/m <sup>3</sup>
Ethanol	87.80	J			ppbv
Formaldehyde	182.00	B	63.60	B	ug/m <sup>3</sup>
Heptanal	78.80		50.70		ug/m <sup>3</sup>
Hexanal	134.00		57.30		ug/m <sup>3</sup>
Methylene Chloride	37.60		435.00		ppbv
m-Tolualdehyde	12.70		33.40		ug/m <sup>3</sup>
NG	1,490.00				ug/m <sup>3</sup>
Nitrous Oxide	766.00		857.00		ppmv
Nonanal	40.70		1.90	U	ug/m <sup>3</sup>
NOx	1.40		1.00		ppmv
Octanal	35.40		0.77	U	ug/m <sup>3</sup>
Octane	80.47			J	ppbv
Oxygen	21.20		21.10		%
Pentanal	0.54	U	32.10		ug/m <sup>3</sup>
Toluene	37.50		60.00	u	ppbv
Total Hydrocarbons	103.00		321.00		ppmv

J = Estimated Value; concentration is below limit of quantification

MAX = Reported result was from a multi-fraction gas sampling train that contains both non-detected results and positive results.

U = Analyte was not detected

D = Result obtained from analysis of a dilution or surrogate diluted below detection limit

B = When applied to anions or organic analysis the qualifier indicates that the analyte was detected in the associated method/instrument blank



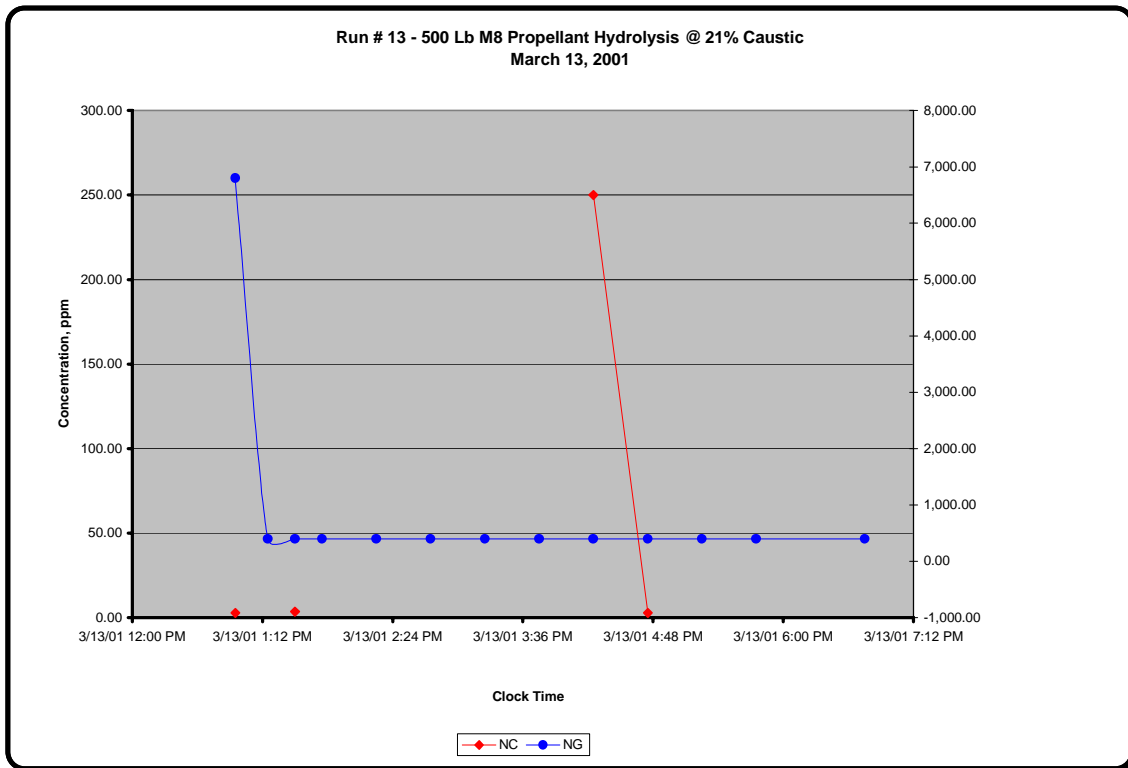


Figure 6-26. Run 13, Mid-Run Destruction Efficiency

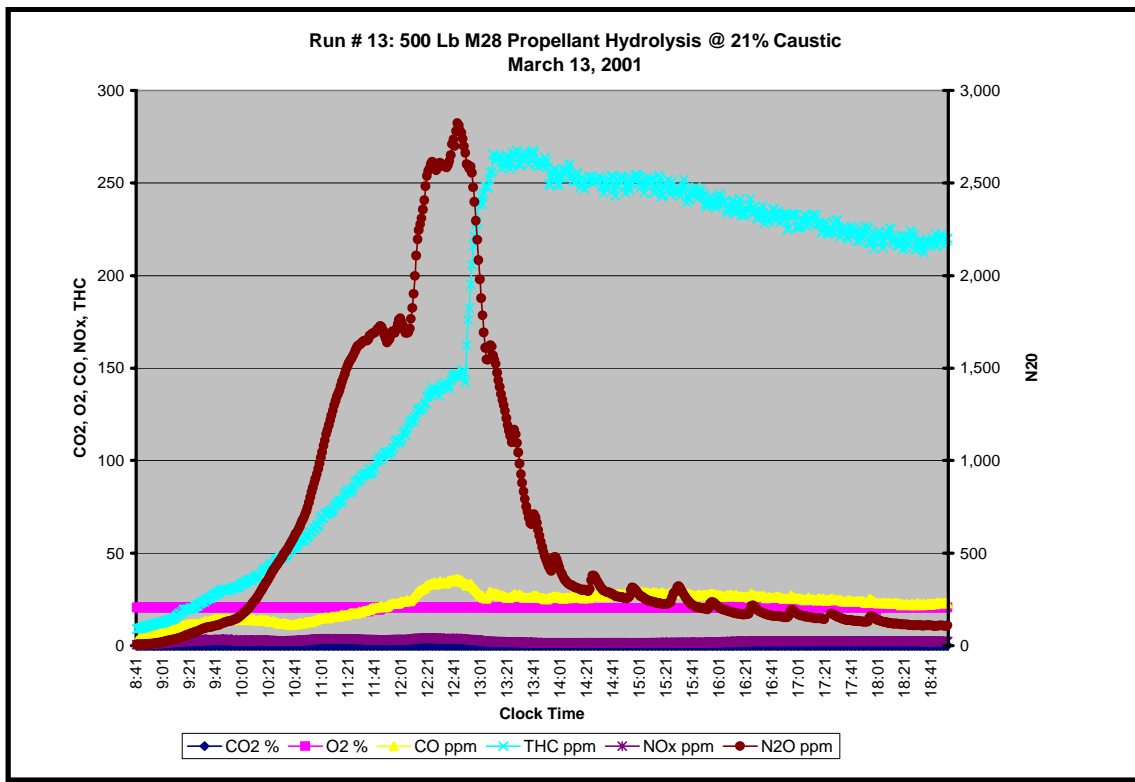


Figure 6-27. Run 13, Off-gas Production

Table 6-18. Run 13, M28 Propellant (Unleaded) End of Run Hydrolysate Characterization

End of Run Hydrolysate Analysis				
Component	Concentration	Unit	ppm	Note
Acetate	10,100.00	mg/l	10,100.00	
Aluminum	236.00	ug/l	0.24	J
Ammonia	95.50	mg/l	95.50	
Barium	124.00	ug/l	0.12	
bis(2-Ethylhexyl)phthalate	330.00	ug/l	0.33	J
Calcium	34,800.00	ug/l	34.80	
Carbazole	610.00	ug/l	0.61	J
Chloride	304.00	mg/l	304.00	
Chromium	195.00	ug/l	0.20	
Cobalt	78.40	ug/l	0.08	
Copper	1,310.00	ug/l	1.31	
Cyanide (Sodium Cyanide)	324,000.00	ug/l	324.00	
Fluoride	1,060.00	mg/l	1,060.00	
Formate	2,440.00	mg/l	2,440.00	
Iron	4,970.00	ug/l	4.97	
Magnesium	7,160.00	ug/l	7.16	
Manganese	54.70	ug/l	0.05	
Molybdenum	72.90	ug/l	0.07	
Nickel	380.00	ug/l	0.38	
Nitrate-N	2,070.00	mg/l	2,070.00	
Nitrite-N	4,930.00	mg/l	4,930.00	
N-Nitrosodiphenylamine	1,900.00	ug/l	1.90	J
o-Phosphate-P	488.00	mg/l	488.00	
Phosphorus	92.30	ug/l	0.09	J
Potassium	18,700.00	ug/l	18.70	
Sodium	106,000,000.00	ug/l	106,000.00	
Sulfate	162.00	mg/l	162.00	
Zinc	348.00	ug/l	0.35	
TIC	1,325.00	mg/l	1,325.00	
TOC	17,200.00	mg/l	17,200.00	
COD	36,300.00	mg/l	36,300.00	
Total Suspended Solids	470.00	mg/l	470.00	
Total Dissolved Solids	262,000.00	mg/l	262,000.00	
Normality as NaOH	3.80	n		
Density	1.19	g/ml		

J = Estimated Value; concentration is below limit of quantification

Table 6-19. Run 13, M28 Propellant (Unleaded) Off-gas Characterization

Reactor Off Gas Analysis					
Component	During Energetic Addition	Note	During Reaction	Note	Unit
1-Butanol	77.73	J			ppbv
2-Butanone	850.00		1,140.00	U	ppbv
Acetaldehyde	10,300.00	D	6,090.00	D	ug/m <sup>3</sup>
Acetone	22,000.00		10,600.00		ppbv
Ammonia	198,000.00		1,120,000.00		ug/m <sup>3</sup>
Benzene	602.00		174.00		ppbv
Bromodichloromethane	22.20		30.00	U	ppbv
Butanal	214.00		407.00		ug/m <sup>3</sup>
Carbon Dioxide	0.02		0.06		%
Carbon Monoxide	10.00		19.00		ppmv
Chloroform	33.00		30.00	U	ppbv
Cyanide	0.00	U	0.04		mg/m <sup>3</sup>
Cyclohexanone	63.50		47.10		ug/m <sup>3</sup>
Dibromochloromethane	13.10		30.00	U	ppbv
Formaldehyde	66.50	B	20.20	B	ug/m <sup>3</sup>
Heptanal	46.90		21.60		ug/m <sup>3</sup>
Hexanal	136.00		25.60		ug/m <sup>3</sup>
Methylene Chloride	214.00	B	266.00	B	ppbv
m-Tolualdehyde	3.15	J	10.90		ug/m <sup>3</sup>
NG	4,030.00				ug/m <sup>3</sup>
Nitrous Oxide	978.00		401.00		ppmv
Nonanal	42.70		1.31	U	ug/m <sup>3</sup>
NOx	2.10		1.20		ppmv
Octanal	35.40		0.53	U	ug/m <sup>3</sup>
Oxygen	21.20		21.10		%
Phenol			18.90	J	ppbv
Toluene	11.50	U	85.50		ppbv
Total Hydrocarbons	95.70		334.70		ppmv

J = Estimated Value; concentration is below limit of quantification.

MAX = Reported result was from a multi-fraction gas sampling train that contains both non-detected results and positive results.

U = Analyte was not detected.

D = Result obtained from analysis of a dilution or surrogate diluted below detection limit.

B = When applied to anions or organic analysis the qualifier indicates that the analyte was detected in the associated method/instrument blank.

### 6.3.2 Discussion & Analysis:

1. The end of run liquid analyses indicates that the desired 100.00% destruction efficiency was achieved approximately 3 hours after cessation of the feeding of M28 propellant to the reactor – 7 hours after the start of the run.
2. The energetics loading for the four runs with M28 unleaded propellant is 7.1 – 7.9 weight-percent resulting in a total solids (dissolved and suspended) loading at the end of the run of between 15.4-to-26.3 weight-percent depending on the caustic strength.
3. The only “bad actor” detected in the hydrolysate is cyanide (possibly sodium cyanide) at a range of 208 to 324 ppm, increasing in concentration with increasing caustic strength. As stated in paragraph 6.1.2 above, the hydrolysis of energetics will produce cyanide and that the subsequent treatment of said hydrolysate using SCOW technology will adequately reduce the hazardous compound concentrations in the hydrolysate feeds well below levels of concern.
4. The airflow across of the reactor headspace was maintained at ~40 scfm during the test. With the exception of THCs, the off gassing quickly fell-off after the addition of the propellant was completed (see Figures 6-25 and 6-27), indicating that the reaction was progressing to conclusion in line with the liquid analysis data. It was conjectured that the THC off gassing was the results of dissolved gasses being liberated and the continued reaction of the caustic solution with the by-products of the destruction of the NC chain.
5. The heat released by the exothermic reaction easily controlled by the reactor jacket cooling system, and the hydrolysate was maintained at the 87°C set point without difficulty.
6. The operating level in the reactor was maintained just above the lower impeller representing a starting volume of ~700-gallons. Foaming was not experienced with M28 propellant.
7. Examination of the off-gas characterization for the two runs indicates that NG vapor was detected in the gas stream only during the addition phase of the process. NG has a measurable vapor pressure at ambient, therefore one would expect NG to come-off as part of the off-gassing stream. Use of a condenser to drop any entrained materials back into the reactor should be effective; however, the condenser should be designed for potential service with NG vapors and cleaned accordingly when the system is decommissioned from service.
8. The major constituents of the off gassing were acetaldehyde, acetone, and ammonia. These gasses can be effectively treated with a scrubber system, with the effluent water from the scrubber then processed through the SCWO system as the final treatment step before release. The average concentration of CO<sub>2</sub>, O<sub>2</sub>, CO, THC, NO<sub>x</sub> and N<sub>2</sub>O in the off gas stream during energetic addition for the two runs was: 0.04%, 21.2%, 34 ppmvd, 103 ppmvd, 1.4 ppmvd, 766 ppmvd, respectively and during digestion the concentration was: 0.05%, 21.1%, 45 ppmvd, 321 ppmvd, 1.0 ppmvd, 857 ppmvd, respectively.
9. The inorganic materials (metals) detected in the hydrolysate end of runs analysis are sourced from the sodium hydroxide stock feed that contains some of these components.

#### 6.4 Tetrytol Explosive Hydrolysis Tests & Results (Runs 15, 16, and 17).

The objective of these tests is to clearly determine and define the optimum operating parameters for the Tetrytol and Tetrytol explosive hydrolysis process to support the design and installation of the full-scale hydrolysis process at the Pueblo and the Blue Grass Chemical Agent Disposal Facility for the destruction of explosives contained in the 4.2-inch Cartridge, Chemical Agent HT and HD, M2 and M2A1; 105mm Projectile, Chemical Agent HD, M60; and 155mm Projectile, Chemical Agent HD, M104 and M110.

The table below identifies the process operating parameters for Runs 15, 16 and 17. Each test was conducted with 350-pounds of Tetrytol as opposed to 500-pounds due to limited supplies over a three hour addition period.

Table 6-20. Tetrytol Explosive Test Parameters

Operating Condition	Run 15	Run 16	Run 17
Tetrytol Feed Rate, lbs/hr			
1 <sup>st</sup> hour	50	50	50
2 <sup>nd</sup> hour	100	100	100
3 <sup>rd</sup> hour	200	200	200
Caustic Soda Concentration, wt%	12	21	26
Caustic Soda Feed, gal	700	700	700
Reactor Operating Temperature, °C	87	87	87
Agitation Speed, RPM	80	80	80
Date Conducted	3/26/2001	3/28/2001	3/30/2001

The following charts represent the process operating and control parameters for the hydrolysis for Tetrytol explosives:

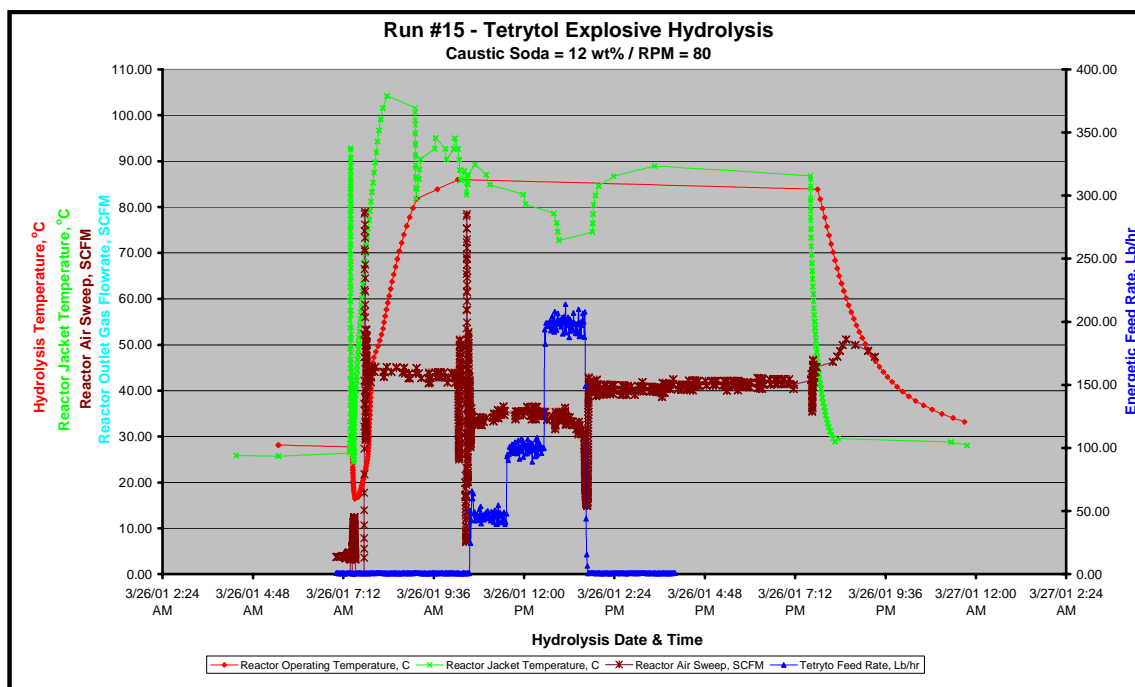


Figure 6-28. Run 15, Process Operating and Control Parameters

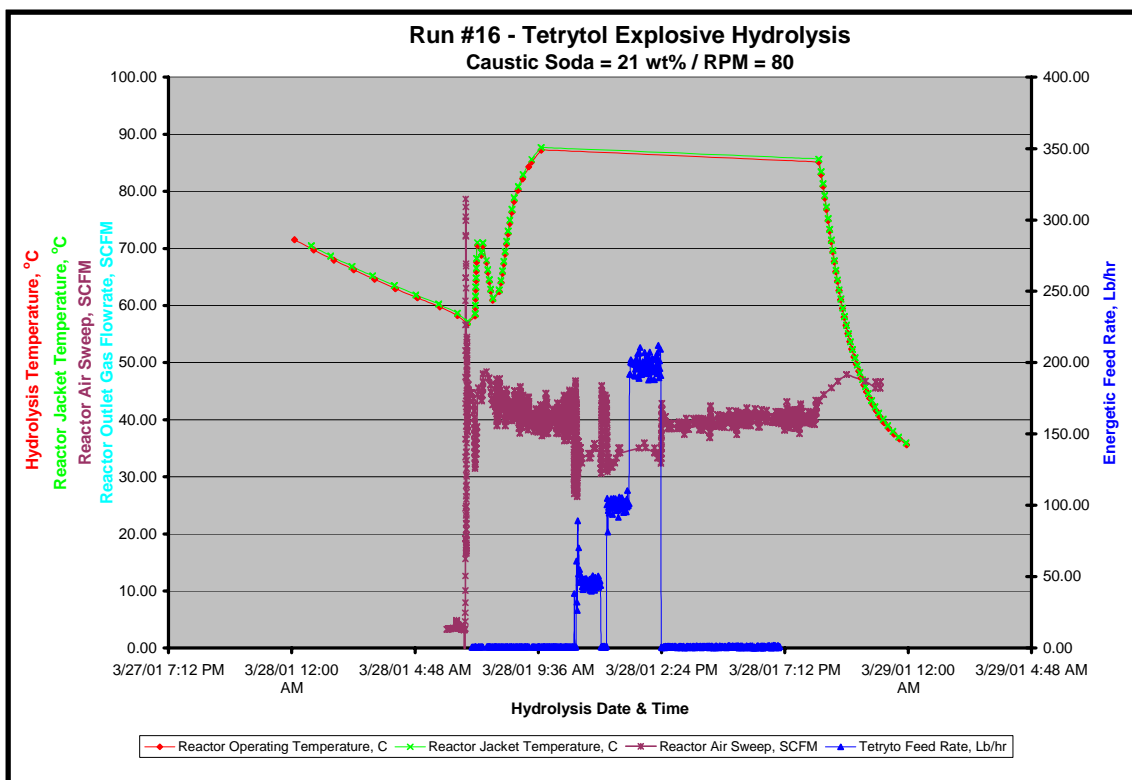


Figure 6-29. Run 16, Process Operating and Control Parameters

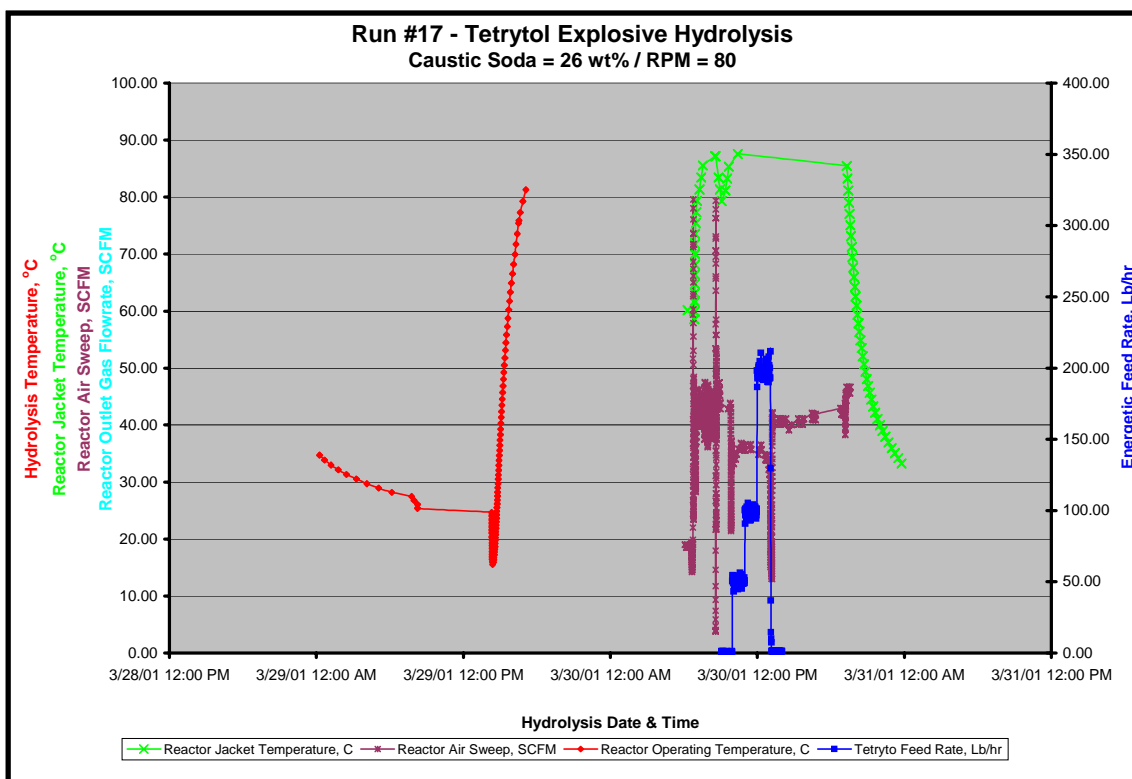


Figure 6-30. Run 17, Process Operating and Control Parameters

#### 6.4.1 Tests Results:

The following plots represent the destruction of Tetrytol explosive as a function of reactor residence time.

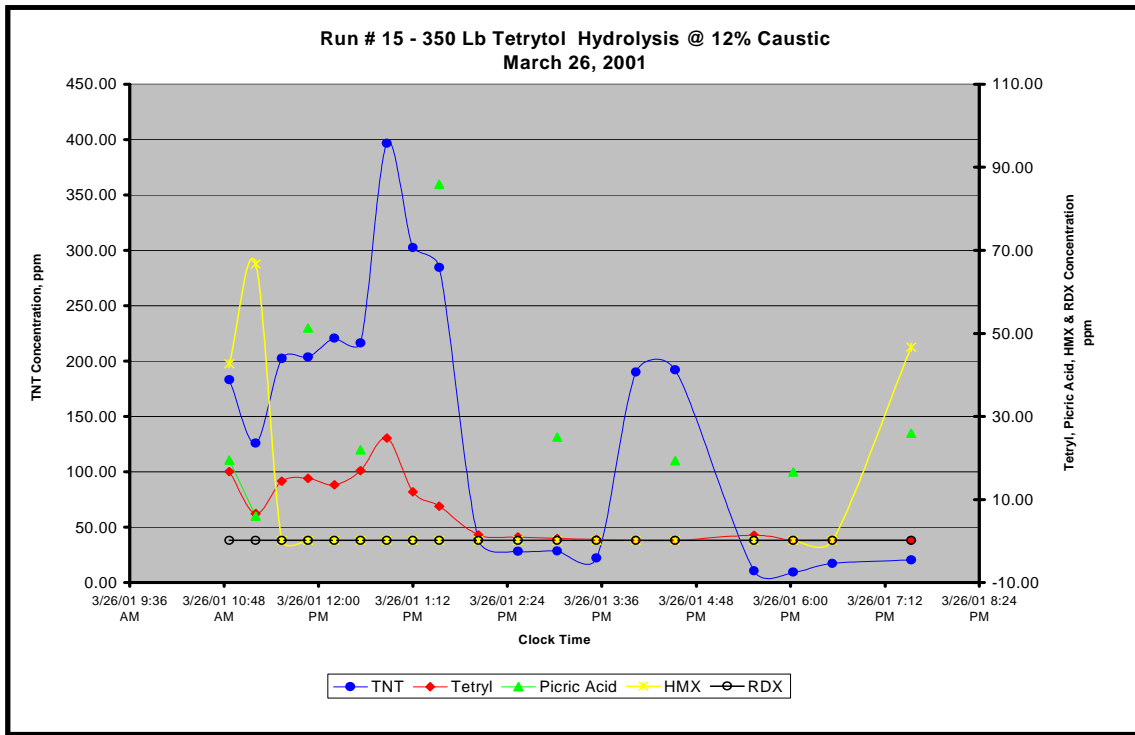


Figure 6-31. Run 15, Mid-Run Destruction Efficiency

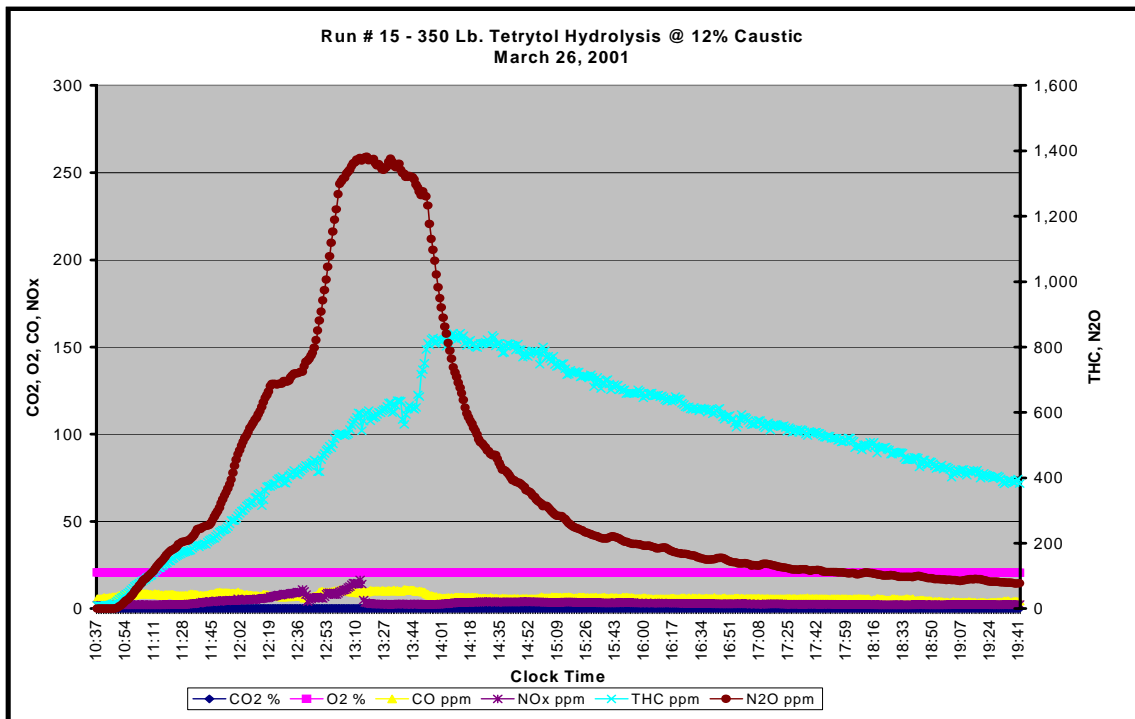


Figure 6-32. Run 15, Off-gas Production

Table 6-21. Run 15, Tetrytol Explosive End of Run Hydrolysate Characterization

End of Run Hydrolysate Analysis				
Component	Concentration	Unit	ppm	Note
2-Fluoro-4-nitrophenol	1,100.00	ug/l	1.10	J
Acetate	438.00	mg/l	438.00	
Ammonia	1,260.00	mg/l	1,260.00	
Calcium	28,300.00	ug/l	28.30	
Cyanide (Sodium Cyanide)	259,000.00	ug/l	259.00	
Diethylphthalate	310.00	ug/l	0.31	J
Formate	2,250.00	mg/l	2,250.00	
Magnesium	7,310.00	ug/l	7.31	
Nitrate-N	29.60	mg/l	29.60	
Nitrite-N	3,460.00	mg/l	3,460.00	
o-Phosphate-P	16.10	mg/l	16.10	
Phosphorus	770.00	ug/l	0.77	J
Sodium	51,300,000.00	ug/l	51,300.00	
Sulfate	400.00	mg/l	400.00	
Zinc	279.00	ug/l	0.28	
TIC	2,235.00	mg/l	2,235.00	
TOC	11,342.50	mg/l	11,342.50	
COD	30,800.00	mg/l	30,800.00	
Total Suspended Solids	200.00	mg/l	200.00	
Total Dissolved Solids	213,000.00	mg/l	213,000.00	
Normality as NaOH	1.60	n		
Density	1.10	g/ml		

J = Estimated Value; concentration is below limit of quantification



Table 6-22. Run 15, Tetrytol Explosive Off-gas Characterization

Reactor Off Gas Analysis					
Component	During Energetic Addition	Note	During Reaction	Note	Unit
1,3,5-Trinitrobenzene	52.2000	MAX			ug/m <sup>3</sup>
1,3-Dinitrobenzene	0.3880	MAX			ug/m <sup>3</sup>
1-Butanol	1,198.5000	J	228.1000	J	ppbv
2,4,6-Trinitrotoluene	80.7000	MAX			ug/m <sup>3</sup>
2,4-Dinitrotoluene	15.2000	MAX			ug/m <sup>3</sup>
2,6-Dinitrotoluene	0.1240	MAX			ug/m <sup>3</sup>
2-Amino-4,6-Dinitrotoluene	0.4350	MAX			ug/m <sup>3</sup>
4-Amino-2,6-Dinitrotoluene	0.6810	MAX			ug/m <sup>3</sup>
Acetaldehyde	10.6000	B	8.0200	B	ug/m <sup>3</sup>
Acetone	1,980.0000	B	2,580.0000	B	ppbv
Ammonia	46,000.0000		178,000.0000		ug/m <sup>3</sup>
Bromomethane	164.0000		112.0000		ppbv
Butanal	0.0971	J	0.1970		ug/m <sup>3</sup>
Carbon Dioxide	0.0000		0.0000		%
Carbon Monoxide	3.0000		0.0000		ppmv
Chloroform	37.3000		15.5000	U	ppbv
Chloromethane	27.5000		25.2000		ppbv
Crotonaldehyde	0.3250	J	0.0534	J	ug/m <sup>3</sup>
Cyanide	0.0015		0.0005		ug/m <sup>3</sup>
Cyclohexanone	0.2050	J	0.0021	U	ug/m <sup>3</sup>
Ethylenimine			677.9000	J	ppbv
Formaldehyde	49.1000	J	63.6000	J	ug/m <sup>3</sup>
Heptanal	0.0883	J	0.0049	U	ug/m <sup>3</sup>
Hexanal	0.1730	J	0.0063	U	ug/m <sup>3</sup>
Methyl Chloride	116.0000	B	23.3000	B	ppbv
m-Tolualdehyde	4.2200		0.1490	J	ug/m <sup>3</sup>
Nitrous Oxide	634.0000		248.0000		ppmv
Nonanal	0.1400	J	0.0329	J	ug/m <sup>3</sup>
NOx	2.8000		0.7000		ppmv
Octanal	0.0852	J	0.0161	J	ug/m <sup>3</sup>
Oxygen	21.4000		21.2000		%
Pentanal	1.0100		0.0035	U	ug/m <sup>3</sup>
Tetryl	14.0000	MAX			ug/m <sup>3</sup>
Toluene	49.8000		39.1000		ppbv
Total Hydrocarbons	492.0000		915.0000		ppmv

J = Estimated Value; concentration is below limit of quantification.

MAX = Reported result was from a multi-fraction gas sampling train that contains both non-detected results and positive results.

U = Analyte was not detected.

D = Result obtained from analysis of a dilution or surrogate diluted below detection limit.

B = When applied to anions or organic analysis the qualifier indicates that the analyte was detected in the associated method/instrument blank.

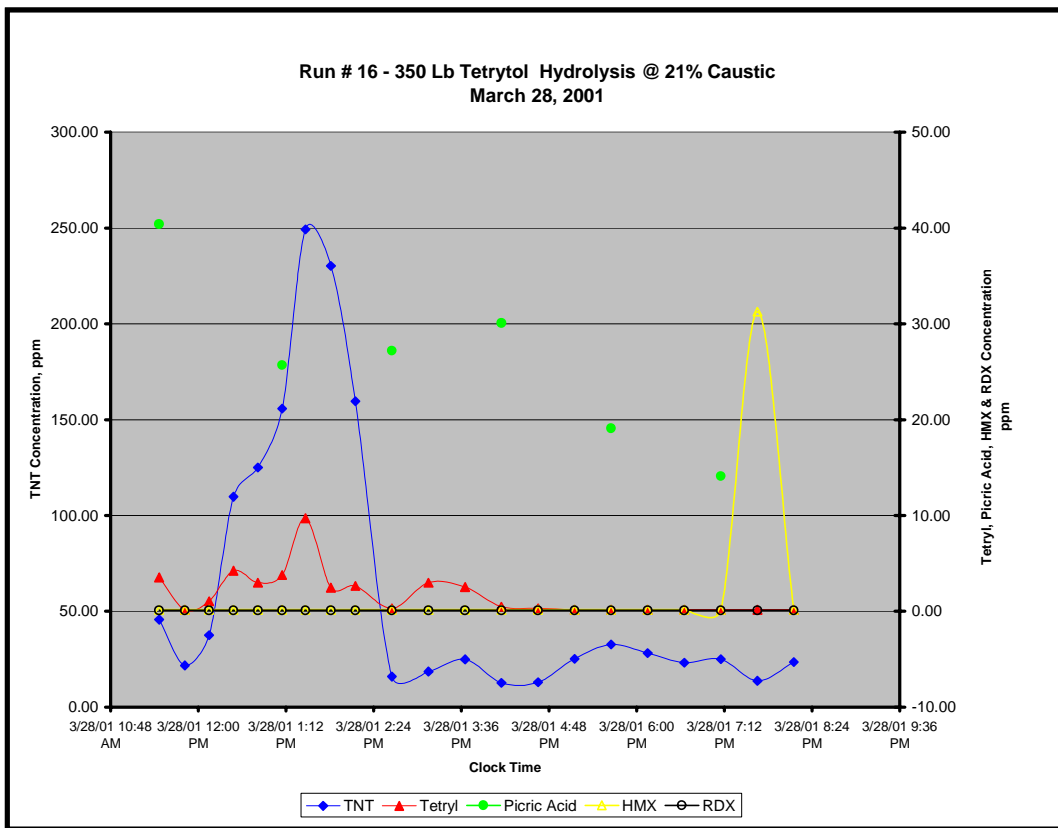


Figure 6-33. Run 16, Mid-Run Composition B4 Destruction Efficiency

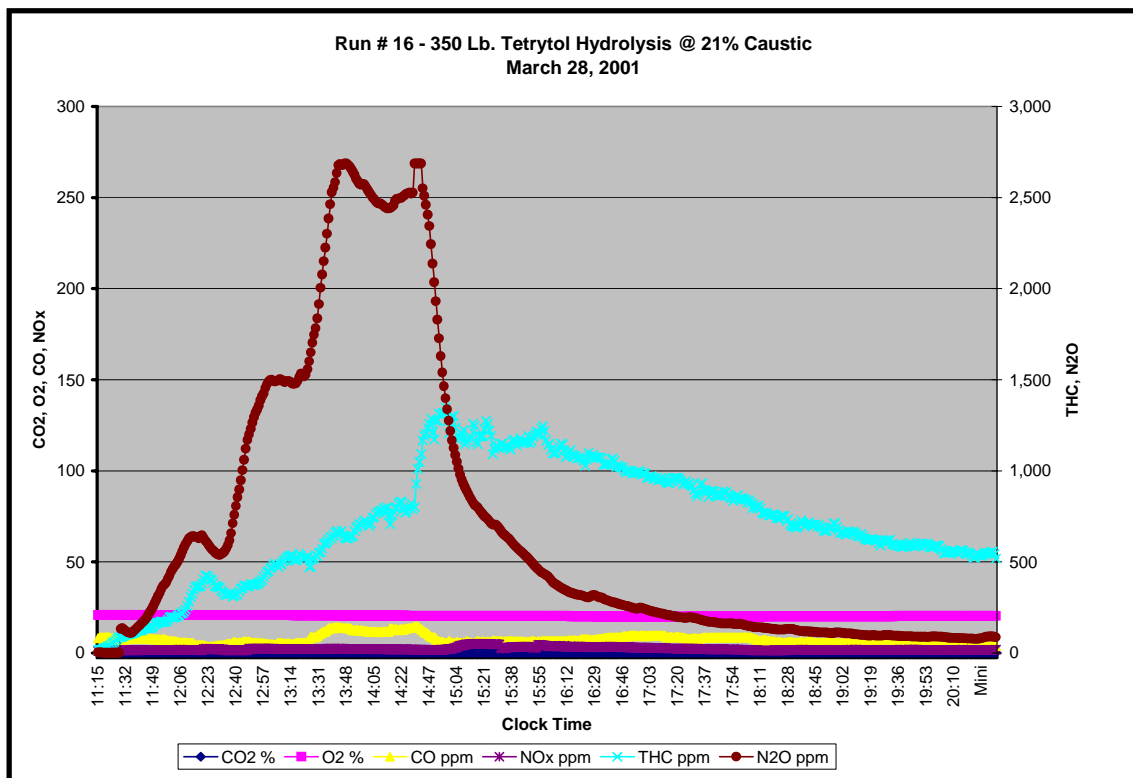


Figure 6-34. Run 16, Off-gas Production

Table 6-23. Run 16, Tetrytol Explosive End of Run Hydrolysate Characterization

End of Run Hydrolysate Analysis				
Component	Concentration	Unit	ppm	Note
2-Fluoro-4-nitrophenol	1,400.00	ug/l	1.40	J
Acetate	566.00	mg/l	566.00	
Ammonia	1,270.00	mg/l	1,270.00	
bis(2-Ethylhexyl)phthalate	280.00	ug/l	0.28	J
Calcium	21,600.00	ug/l	21.60	
Cyanide (Sodium Cycnide)	315,000.00	ug/l	315.00	
Diethylphthalate	300.00	ug/l	0.30	J
Formate	2,930.00	mg/l	2,930.00	
HMX	28,495.00	ug/l	28.50	
Magnesium	6,830.00	ug/l	6.83	
Nitrate-N	44.60	mg/l	44.60	
Nitrite-N	3,210.00	mg/l	3,210.00	
o-Phosphate-P	29.00	mg/l	29.00	
Phosphorus	815.00	ug/l	0.82	J
Picric Acid	73.10	mg/l	73.10	
Potassium	180,000.00	ug/l	180.00	
Sodium	102,000,000.00	ug/l	102,000.00	
Sulfate	476.00	mg/l	476.00	
TNT	14,397.00	ug/l	14.40	
Zinc	315.00	ug/l	0.32	
TIC	2,135.00	mg/l	2,135.00	
TOC	12,475.00	mg/l	12,475.00	
COD	30,200.00	mg/l	30,200.00	
Total Suspended Solids	200.00	mg/l	200.00	
Total Dissolved Solids	251,000.00	mg/l	251,000.00	
Normality as NaOH	3.20	n		
Density	1.21	g/ml		

J = Estimated Value; concentration is below limit of quantification

Table 6-24. Run 16, Tetrytol Explosive Off-gas Characterization

Reactor Off Gas Analysis					
Component	During Energetic Addition	Note	During Reaction	Note	Unit
1,3,5-Trinitrobenzene	0.5540	MAX			ug/m <sup>3</sup>
1,3-Dinitrobenzene	0.5430	MAX			ug/m <sup>3</sup>
2,4,6-Trinitrotoluene	80.0000	MAX			ug/m <sup>3</sup>
2,4-Dinitrotoluene	12.4000	MAX			ug/m <sup>3</sup>
2,6-Dinitrotoluene	0.1260	MAX			ug/m <sup>3</sup>
2-Amino-4,6-Dinitrotoluene	0.3630	MAX			ug/m <sup>3</sup>
4-Amino-2,6-Dinitrotoluene	0.5090	MAX			ug/m <sup>3</sup>
Acetaldehyde	12.2000	B	1.7800	B	ug/m <sup>3</sup>
Acetone	1,690.0000	B	1,030.0000		ppbv
Ammonia	59,100.0000		221,000.0000		ug/m <sup>3</sup>
Bromomethane	52.8000		22.5000	U	ppbv
Butanal	0.3950		0.0394	J	ug/m <sup>3</sup>
Carbon Dioxide	0.0000		0.0000		%
Carbon Monoxide	0.0000		0.0000		ppmv
Chloroform	40.4000		15.0000	U	ppbv
Crotonaldehyde	0.4100		0.1560		ug/m <sup>3</sup>
Cyanide	0.0010		0.0004		ug/m <sup>3</sup>
Cyclohexanone	0.0292	J	0.0015	U	ug/m <sup>3</sup>
Ethylenimine			4,020.0000	J	ppbv
Formaldehyde	21.8000	B	18.4000	B	ug/m <sup>3</sup>
Heptanal	0.2670	J	0.0033	U	ug/m <sup>3</sup>
Hexanal	1.9900		0.0662	J	ug/m <sup>3</sup>
Methyl Chloride	30.0000	U	32.6000	B	ppbv
m-Tolualdehyde	0.3320		0.0042		ug/m <sup>3</sup>
Nitrous Oxide	1,279.0000		86.0000		ppmv
Nonanal	0.1630	J	0.0383	J	ug/m <sup>3</sup>
NOx	1.0000		0.9000		ppmv
Octanal	0.1230	J	0.0196	J	ug/m <sup>3</sup>
Oxygen	21.1000		20.8000		%
Pentanal	1.2200		0.0279	J	ug/m <sup>3</sup>
Tetryl	11.3000	MAX			ug/m <sup>3</sup>
Toluene	14.7000		15.0000	U	ppbv
Total Hydrocarbons	601.0000		755.0000		ppmv

J = Estimated Value; concentration is below limit of quantification.

MAX = Reported result was from a multi-fraction gas sampling train that contains both non-detected results and positive results.

U = Analyte was not detected.

D = Result obtained from analysis of a dilution or surrogate diluted below detection limit.

B = When applied to anions or organic analysis the qualifier indicates that the analyte was detected in the associated method/instrument blank

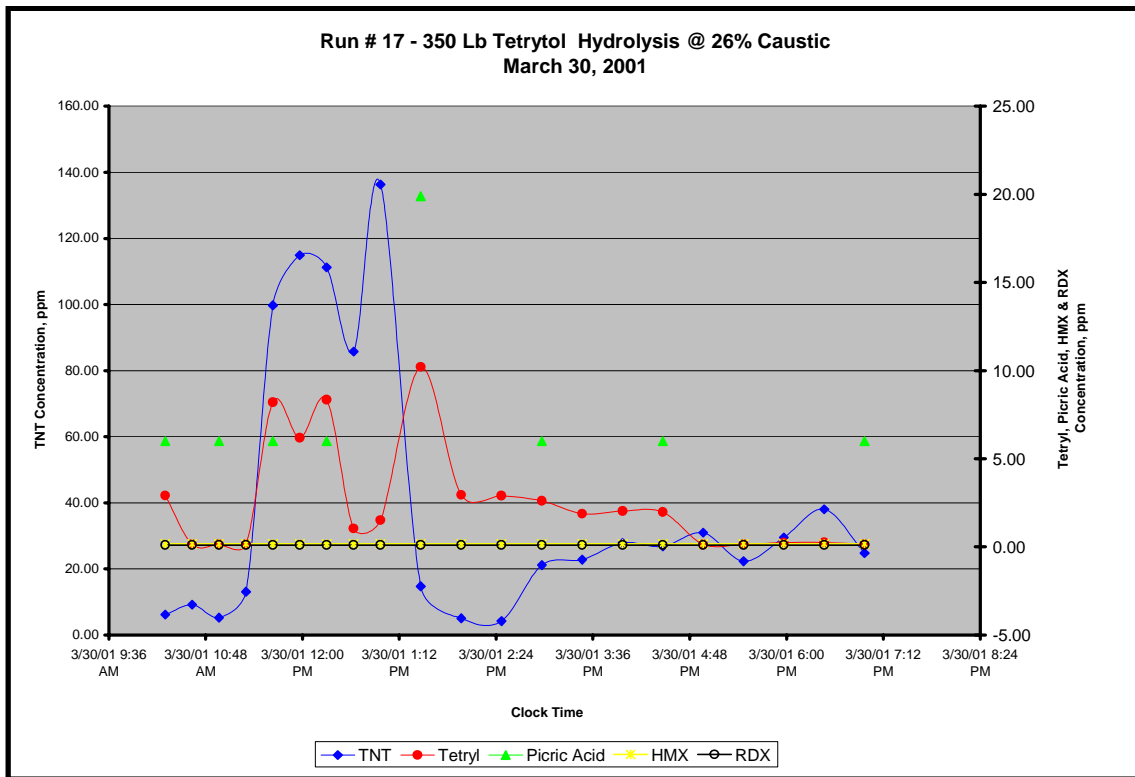


Figure 6-35. Run 17, Mid-Run Tetrytol Explosive Destruction Efficiency

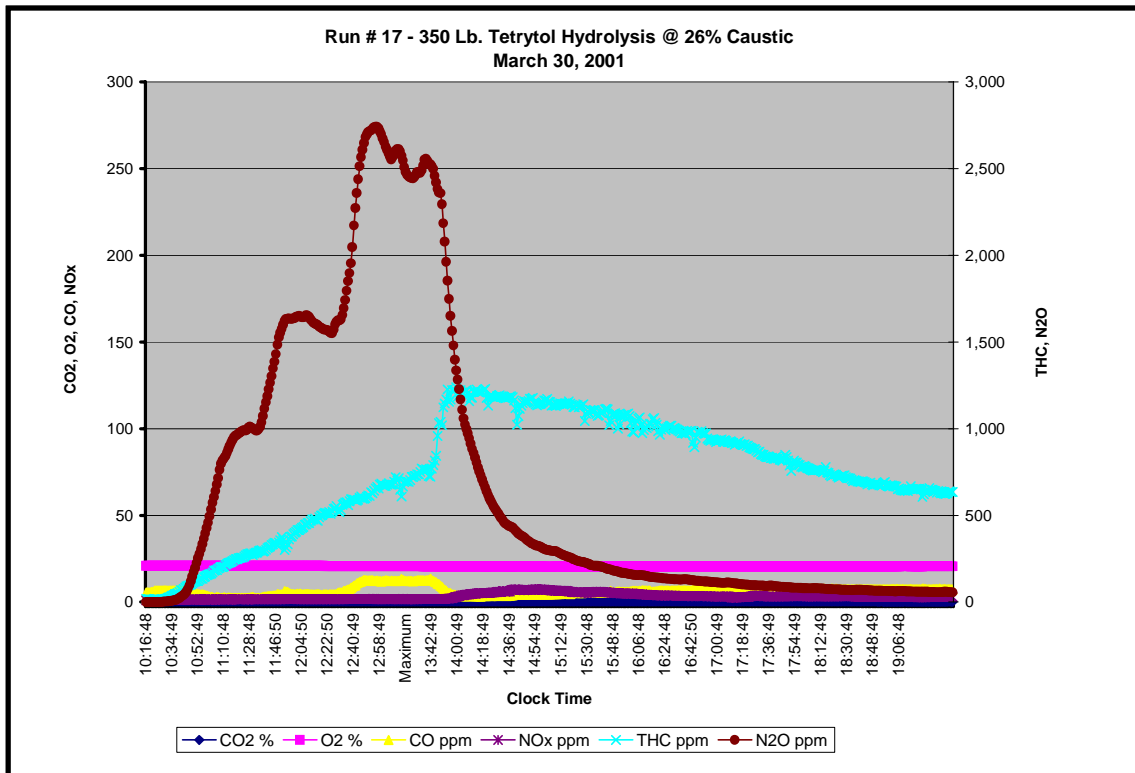


Figure 6-36. Run 17, Off-gas Production

Table 6-25. Run 17, Tetrytol Explosive End of Run Hydrolysate Characterization

End of Run Hydrolysate Analysis				
Component	Concentration	Unit	ppm	Note
Acetate	719.00	mg/l	719.00	
Ammonia	955.00	mg/l	955.00	
Barium	256.00	ug/l	0.26	
Calcium	17,900.00	ug/l	17.90	
Copper	521.00	ug/l	0.52	
Cyanide (Sodium Cyanide)	705,000.00	ug/l	705.00	
Fluoride	1,060.00	mg/l	1,060.00	
Formate	3,660.00	mg/l	3,660.00	
HMX	37,577.00	ug/l	37.58	
Magnesium	6,540.00	ug/l	6.54	
Molybdenum	59.90	ug/l	0.06	
Nitrate-N	30.10	mg/l	30.10	
Nitrite-N	2,740.00	mg/l	2,740.00	
o-Phosphate-P	30.20	mg/l	30.20	
Picric Acid	39.10	mg/l	39.10	
Potassium	122,000.00	ug/l	122.00	
Sodium	116,000,000.00	ug/l	116,000.00	
Sulfate	273.00	mg/l	273.00	
TNT	28,338.00	ug/l	28.34	
Zinc	333.00	ug/l	0.33	
TIC	3,282.50	mg/l	3,282.50	
TOC	10,475.00	mg/l	10,475.00	
COD	31,700.00	mg/l	31,700.00	
Total Suspended Solids	15,900.00	mg/l	15,900.00	
Total Dissolved Solids	318,000.00	mg/l	318,000.00	
Normality as NaOH	5.50	n		
Density	1.28	g/ml		

Table 6-26. Run 17, Tetrytol Explosive Off-gas Characterization

Reactor Off Gas Analysis					
Component	During Energetic Addition	Note	During Reaction	Note	Unit
1,3,5-Trinitrobenzene	0.7250	MAX			ug/m <sup>3</sup>
1,3-Dinitrobenzene	0.8280	MAX			ug/m <sup>3</sup>
2,4,6-Trinitrotoluene	106.0000	MAX			ug/m <sup>3</sup>
2,4-Dinitrotoluene	14.9000	MAX			ug/m <sup>3</sup>
2,6-Dinitrotoluene	0.1860	MAX			ug/m <sup>3</sup>
2-Amino-4,6-Dinitrotoluene	0.2770	MAX			ug/m <sup>3</sup>
4-Amino-2,6-Dinitrotoluene	0.4610	MAX			ug/m <sup>3</sup>
Acetaldehyde	11.6000	B	2.4300	B	ug/m <sup>3</sup>
Acetone	3,030.0000		16.0000	U	ppbv
Ammonia	73,300.0000		303,000.0000		ug/m <sup>3</sup>
Bromomethane	78.7000		41.3000		ppbv
Butanal	0.3600		0.0306	J	ug/m <sup>3</sup>
Carbon Dioxide	0.0200		0.0100		%
Carbon Monoxide	0.0000		0.0000		ppmv
Chloroform	58.2000		16.0000	U	ppbv
Crotonaldehyde	0.1180	J	0.0683	J	ug/m <sup>3</sup>
Cyanide	0.0003		0.0007		ug/m <sup>3</sup>
Cyclohexanone	0.7020	J	0.0264	JB	ug/m <sup>3</sup>
Ethylenimine			12,839.5000	J	ppbv
Formaldehyde	17.8000	B	11.2000	B	ug/m <sup>3</sup>
Heptanal	0.1410	J	0.0048	U	ug/m <sup>3</sup>
Hexanal	1.4700		0.1740	J	ug/m <sup>3</sup>
Methyl Chloride	25.2000	B	24.0000	U	ppbv
m-Tolualdehyde	0.7750		0.0057	U	ug/m <sup>3</sup>
Nitrous Oxide	1,403.0000		276.0000		ppmv
Nonanal	0.1460	J	0.0461	J	ug/m <sup>3</sup>
NOx	0.0000		2.1000		ppmv
Octanal	0.0873	J	0.0258	J	ug/m <sup>3</sup>
Oxygen	21.2000		20.8000		%
Pentanal	1.5400	B	0.0469	JB	ug/m <sup>3</sup>
Tetryl	21.8000	MAX			ug/m <sup>3</sup>
Total Hydrocarbons	540.0000		1,326.0000		ppmv

J = Estimated Value; concentration is below limit of quantification.

MAX = Reported result was from a multi-fraction gas sampling train that contains both non-detected results and positive results.

U = Analyte was not detected.

D = Result obtained from analysis of a dilution or surrogate diluted below detection limit.

B = When applied to anions or organic analysis the qualifier indicates that the analyte was detected in the associated method/instrument blank

#### 6.4.2 Discussion & Analysis:

1. The end of run liquid analyses indicates that the destruction rate efficiency achieved approximately 5 hours after cessation of the feeding of Tetrytol explosive to the reactor – 8 hours after the start of the run – ranged from 100.00% to 99.7847%. The TNT destruction rate efficiency for Runs 16 and 17 was 99.8982% and 99.8063% respectively. Picric Acid was detected during mid-run analysis as shown in Figures 6-31, 6-33 and 6-35, and in the end of run analyses for Runs 16 and 17.
2. The energetics loading for the three runs with Tetrytol is 5.0 – 5.5 weight-percent resulting in a total solids (dissolved and suspended) loading at the end of the run of between 21.3 to 33.3 weight-percent depending on the caustic strength.
3. The only “bad actor” detected in the hydrolysate is cyanide (possibly sodium cyanide) at a range of 259 to 705 ppm, increasing in concentration with increasing caustic strength. As stated in paragraph 6.1.2 above, the hydrolysis of energetics will produced cyanide and that the subsequent treatment of said hydrolysate using SCOW technology adequately reduces the hazardous compound concentrations in the hydrolysate feeds well below levels of concern. Picric acid was detected in the hydrolysate for Runs 16 and 17 at a level of 73 and 39 ppm. The Picric Acid in the hydrolysate is most likely showing up as sodium picrate because the likelihood of finding Picric Acid after more than 8 hours of processing is extremely low.
4. Trace amounts of HMX were detected in the hydrolysate for Runs 16 and 17, which is suspect since the HMX in the Composition B4 runs was 100% destroyed by the caustic. Consequently, it is unclear where the HMX is sourced from (unless it is a by-product from the nitration of Tetryl) and why it remains in the hydrolysate.
5. The airflow across of the reactor headspace was maintained at ~40 scfm during the test. The off gassing fell-off quickly after the addition of the Tetrytol was completed (see Figures 6-32, 6-34, and 6-36), indicating that most of the reactions are taking place during energetic addition and progressing to completion in line with the liquid analysis data.
6. The heat released by the exothermic reaction easily controlled by the reactor jacket cooling system, and the hydrolysate was maintained at the 87°C set point without difficulty.
7. The operating level in the reactor was maintained just above the lower impeller representing a starting volume of ~700-gallons. Foaming was not a problem with Tetrytol explosive.
8. Examination of the off-gas characterization for the three runs indicates that some energetic material (1,3,5-Trinitrobenzene, Dinitrotoluene, TNT, Tetryl) at low level were entrained in the gas stream during the addition phase of the process. TNT has a measurable vapor pressure at ambient, therefore one would expect TNT to come-off as part of the off-gassing stream. Also, the Tetrytol contained significant “fines” and dusting occurs as the Tetrytol falls into the reactor through the headspace air stream, possibly contributing to these readings. The energetic materials disappeared once the addition was stopped. Use of a condenser on top of the reactor to drop any entrained materials back into the reactor should be effective.



9. The major constituents of the off gassing were ammonia, acetone, bromomethane and nitrous oxide with ammonia dominating. These gasses can be effectively treated with a scrubber system, with the water from the scrubber then processed through the SCWO system as the final treatment step before release. The average range concentration of CO<sub>2</sub>, O<sub>2</sub>, CO, THC, NO<sub>x</sub> and N<sub>2</sub>O in the off gas stream during energetic addition for the three runs was: 0.0 - .02%, 21.1 – 21.4%, 0.0 – 3.0 ppmvd, 492 – 601 ppmvd, 0.0 – 2.8 ppmvd, 634 – 1,403 ppmvd, respectively and during digestion the concentration was: 0.0 - 0.01%, 20.8 – 21.2%, 0.0 ppmvd, 755 – 1326 ppmvd, 0.7 – 2.1 ppmvd, 86 – 276 ppmvd, respectively.
10. The inorganic materials (metals) detected in the hydrolysate end of runs analysis are sourced from the sodium hydroxide stock feed that contains some of these components.

## 6.5 M8 Sheet Propellant Hydrolysis Tests & Results (Runs 18, 19, & 20).

The objective of these tests is to clearly determine and define the optimum operating parameters for the M8 sheet propellant hydrolysis process to support the design and installation of the full-scale hydrolysis process at the Pueblo Chemical Agent Disposal Facility for the destruction of propellant contained in the 4.2-inch Cartridge, Chemical Agent HT and HD, M2 and M2A1.

Mid-run liquid samples were not taken during the processing of the M8 propellant because of the configuration of the propellant charge. The M8 propellant is in multiple sheet form, sewn together with cotton threads. Separation of the sheet propellant from the cotton threads was impractical since the rubbery texture of the propellant gripped the threads. The dimensions of the bundles are as follows:

- L & W (maximum): 2.75-inches by 2.75-inches
- Number of sheets: up to 10
- Thickness (per sheet): 0.028-inches, -0.005-inches
- Hole Diameter: 1.31-inches, +0.10-inches

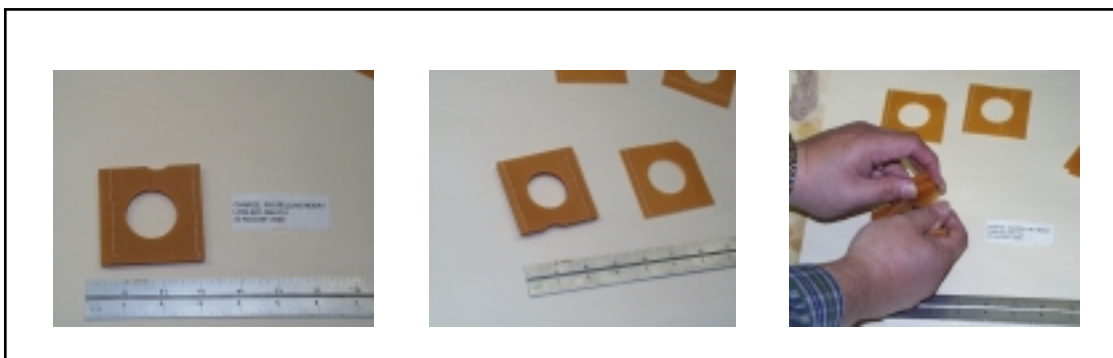


Figure 6-37. M8 Sheet Propellant

Therefore, the M8 propellant was manually fed in the “assembled” condition at the same as stated for the automatic feed rates when using the weigh-feeder system. Consequently, the operating and control parameters charts for the M8 propellant runs do not show the feed rate trace because the weigh-feeder system was not employed. As the sheet propellant was hydrolyzed, the freed cotton threads “blinded” the recirculation line inlet, thereby preventing liquid sampling from taking place during the run.

The table below identifies the process operating parameters for Runs 18, 19 and 20.

Table 6-27. M8 Propellant Test Parameters

Operating Condition	Run 18	Run 19	Run 20
M8 Feed Rate, lbs/hr			
1 <sup>st</sup> hour	50	50	50
2 <sup>nd</sup> hour	100	100	100
3 <sup>rd</sup> hour	150	150	150
4 <sup>th</sup> hour	200	200	200
Caustic Soda Concentration, wt%	12	21	26
Caustic Soda Feed, gal	700	700	700
Reactor Operating Temperature, °C	87	87	87
Agitation Speed, RPM	80	80	80
Date Conducted	4/3/2001	4/12/2001	4/18/2001

The following charts represent the process operating and control parameters for hydrolysis of M8 propellants:

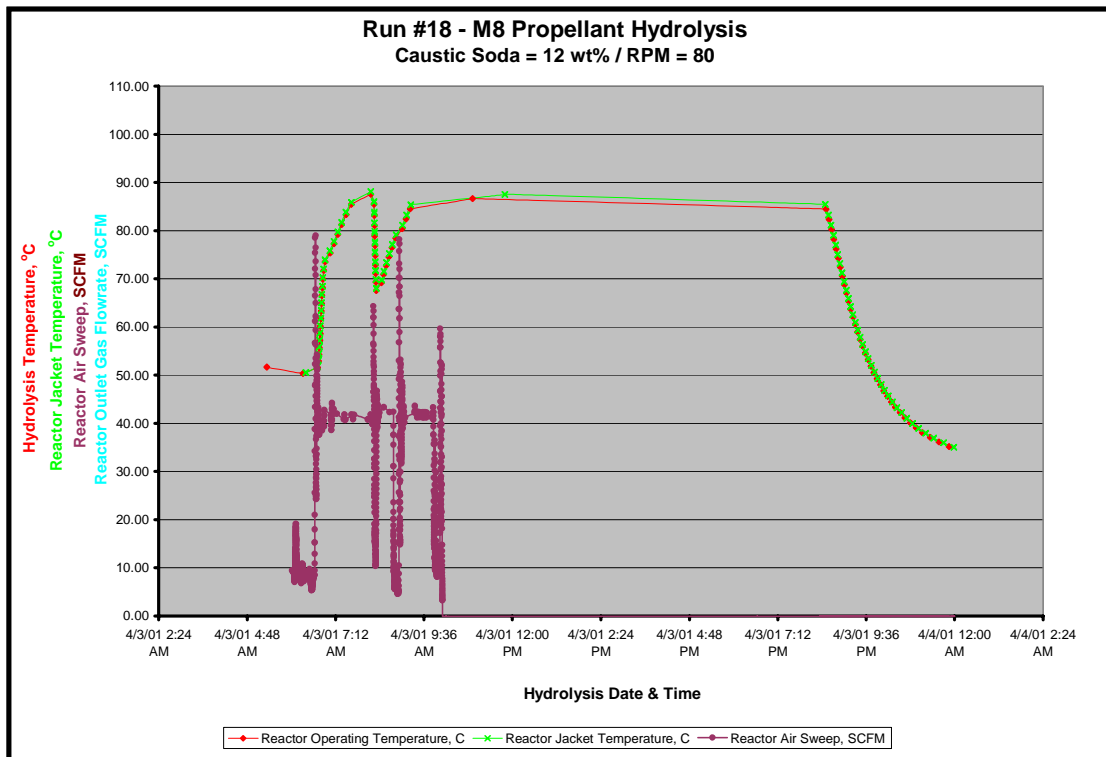


Figure 6-38. Run 18, Process Operating and Control Parameters

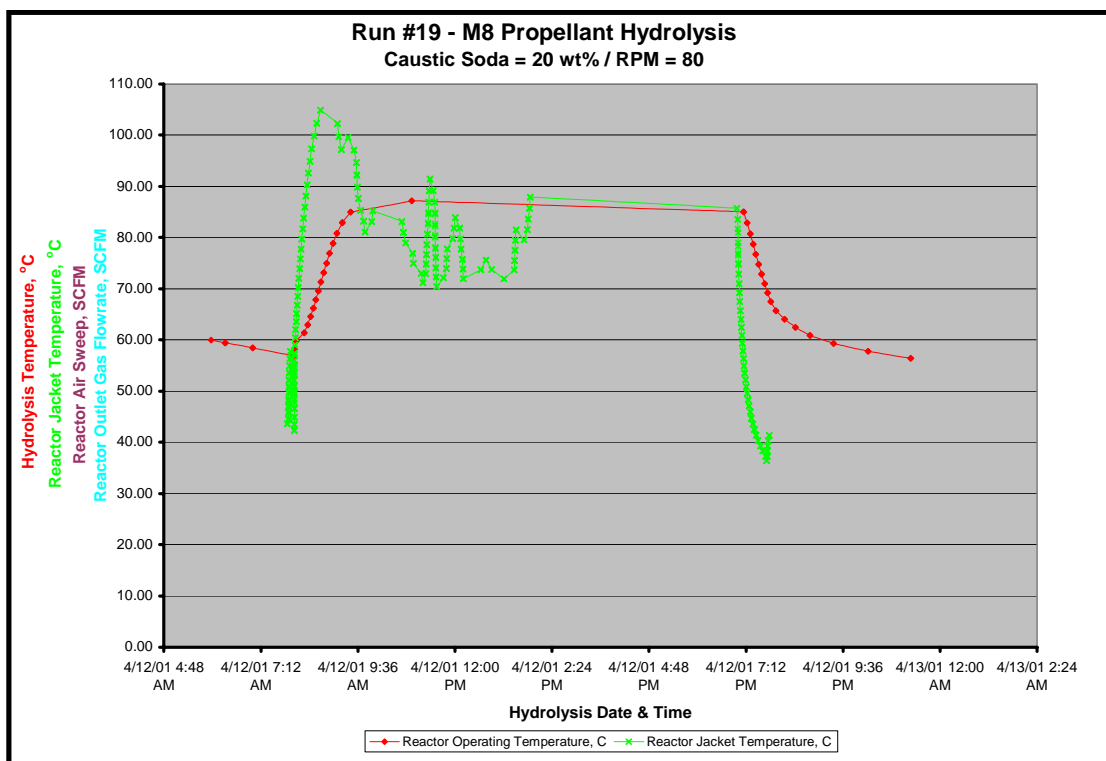


Figure 6-39. Run 19, Process Operating and Control Parameters

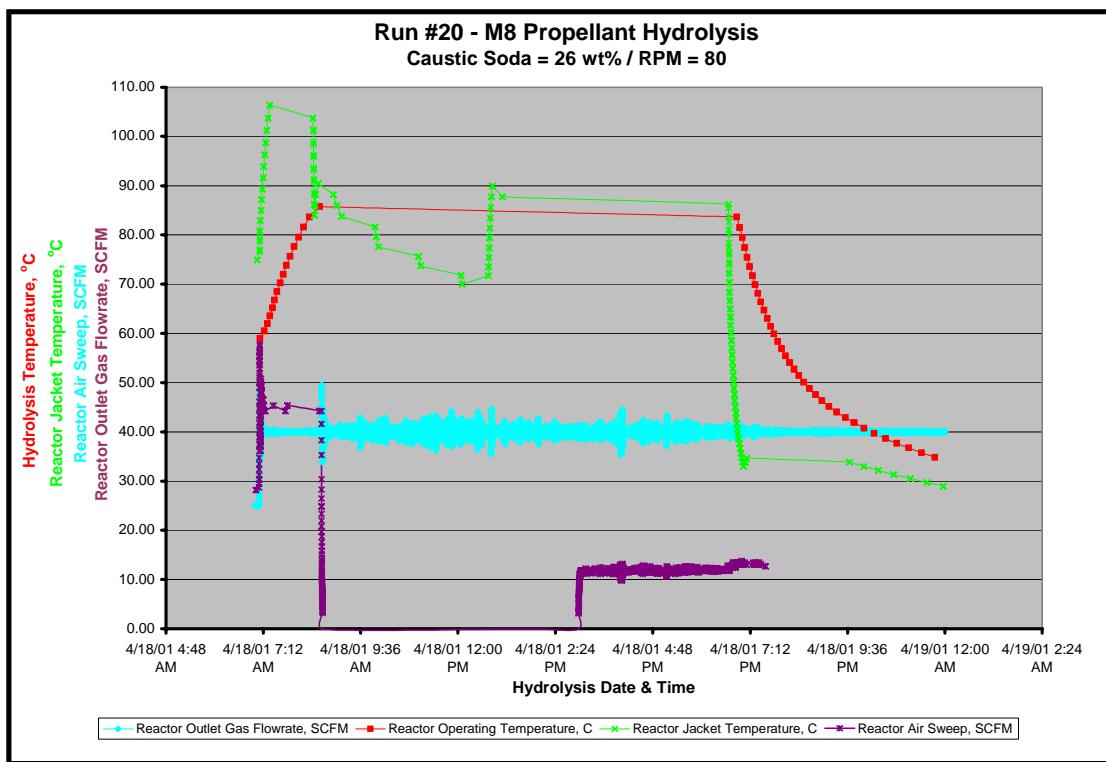


Figure 6-40. Run 20, Process Operating and Control Parameters

#### 6.5.1 Tests Results:

The following plots represent M8 propellant destruction as a function of reactor residence time.

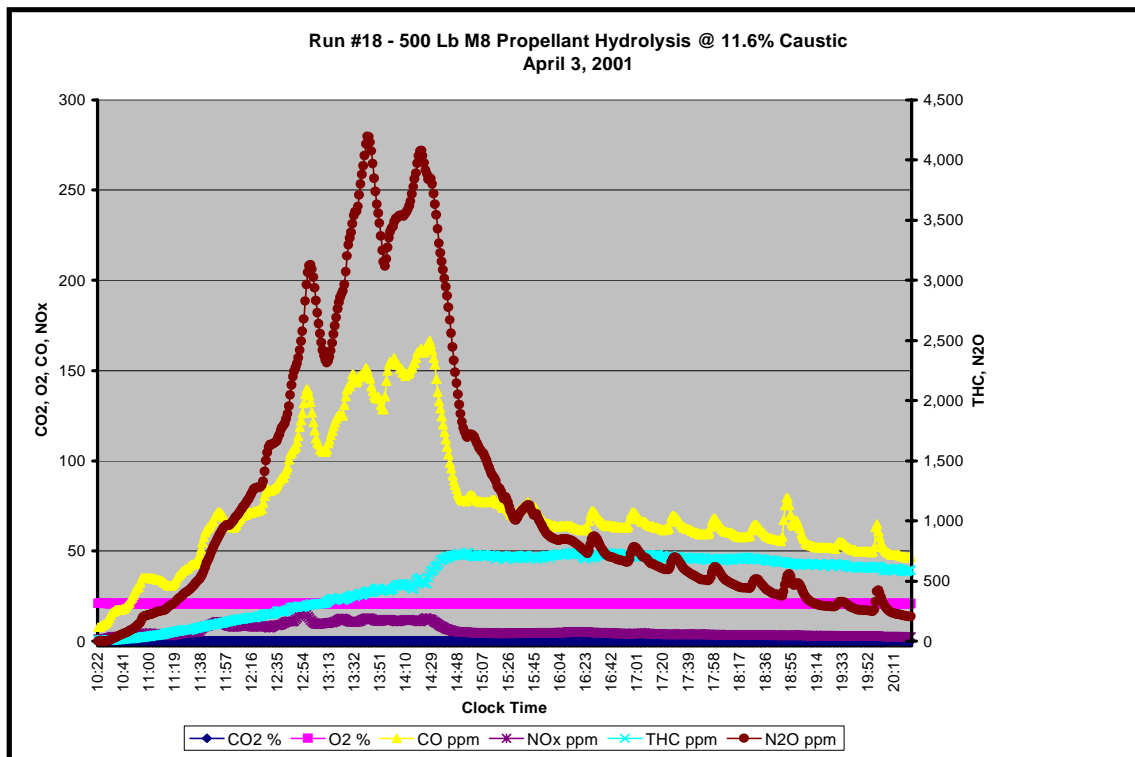


Figure 6-41. Run 18, Off-gas Production

Table 6-28. Run 18, M8 Propellant End of Run Hydrolysate Characterization

End of Run Hydrolysate Analysis				
Component	Concentration	Unit	ppm	Note
1,4:3,6-Dianhydro-.alpha.-d-glucopyranos	1,200.00	ug/l	1.20	J
2-Fluoro-4-nitrophenol	2,800.00	ug/l	2.80	J
Acetate	8,000.00	mg/l	8,000.00	
Aluminum	298.00	ug/l	0.30	
Ammonia	140.00	mg/l	140.00	
Barium	93.10	ug/l	0.09	
bis(2-Ethylhexyl)phthalate	4,300.00	ug/l	4.30	J
Calcium	51,000.00	ug/l	51.00	
Copper	2,100.00	ug/l	2.10	
Cyanide (Sodium Cyanide)	276,000.00	mg/l	276.00	
Fluoride	1,010.00	mg/l	1,010.00	
Formate	2,170.00	mg/l	2,170.00	
Magnesium	7,700.00	ug/l	7.70	
NG	11,199.00	ug/l	11.20	
Nitrate-N	2,560.00	mg/l	2,560.00	
Nitrite-N	6,090.00	mg/l	6,090.00	
Nitrocellulose	0.08	mg/l	0.08	J
Potassium	339,000.00	ug/l	339.00	
Sodium	50,620,000.00	ug/l	50,620.00	
Sulfate	120.00	mg/l	120.00	
Zinc	285.00	ug/l	0.29	
TIC	768.00	mg/l	768.00	
TOC	12,500.00	mg/l	12,500.00	
COD	15,000.00	mg/l	15,000.00	
Total Suspended Solids	132.00	mg/l	132.00	
Total Dissolved Solids	137,000.00	mg/l	137,000.00	
Normality as NaOH	1.29	n		
Density	1.10	g/ml		

J = Estimated Value; concentration is below limit of quantification

Table 6-29. Run 18, M8 Propellant Off-gas Characterization

Reactor Off Gas Analysis					
Component	During Energetic Addition	Note	During Reaction	Note	Unit
2-Butanone	361.00	U	2,220.00		ppbv
Acetaldehyde	6,260.00	D	9,400.00	D	ug/m <sup>3</sup>
Acetone	12,300.00		47,016.00		ppbv
Ammonia	575,000.00		1,850,000.00		ug/m <sup>3</sup>
Benzene	70.20		414.00		ppbv
Bromomethane	14.30	U	23.90		ppbv
Butanal	138.00		830.00		ug/m <sup>3</sup>
Carbon Dioxide	0.04		0.04		%
Carbon Monoxide	79.00		62.00		ppmv
Chloroform	10.70		18.00	U	ppbv
Crotonaldehyde	19.40		21.70		ug/m <sup>3</sup>
Cyanide	0.02		0.15		mg/m <sup>3</sup>
Cyclohexanone	29.30	B	18.30	B	ug/m <sup>3</sup>
Decanal	4.47	J	13.90		ug/m <sup>3</sup>
Ethanol			12,069.00	J	ppbv
Formaldehyde	282.00	B	246.00	B	ug/m <sup>3</sup>
Heptanal	3.38	J	8.69	J	ug/m <sup>3</sup>
Hexanal	36.30		29.70		ug/m <sup>3</sup>
Methylene Chloride	2,150.00	B	27.00	U	ppbv
NG	1,250.00				ug/m <sup>3</sup>
Nitrous Oxide	1,712.00		855.00		ppmv
Nonanal	43.00		5.29	J	ug/m <sup>3</sup>
NOx	7.30		3.10		ppmv
Octanal	3.81	J	7.37	J	ug/m <sup>3</sup>
Oxygen	21.10		21.10		%
Pentanal	26.00	B	46.30	B	ug/m <sup>3</sup>
Toluene	509.00		18.00	U	ppbv
Total Hydrocarbons	342.00		1,016.00		ppmv

J = Estimated Value; concentration is below limit of quantification.

MAX = Reported result was from a multi-fraction gas sampling train that contains both non-detected results and positive results.

U = Analyte was not detected.

D = Result obtained from analysis of a dilution or surrogate diluted below detection limit.

B = When applied to anions or organic analysis the qualifier indicates that the analyte was detected in the associated method/instrument blank.

Table 6-30. Run 19, M8 Propellant End of Run Hydrolysate Characterization

End of Run Hydrolysate Analysis				
Component	Concentration	Unit	ppm	Note
Acetate	10,000.00	mg/l	10,000.00	
Ammonia	154.00	mg/l	154.00	
Barium	168.00	ug/l	0.17	
Calcium	30,800.00	ug/l	30.80	
Chromium	167.00	ug/l	0.17	
Cobalt	106.00	ug/l	0.11	
Cobalt	48.00	ug/l	0.05	J
Copper	1,390.00	ug/l	1.39	
Cyanide (Sodium Cyanide)	378,000.00	ug/l	378.00	
Fluoride	622.00	mg/l	622.00	
Formate	3,050.00	mg/l	3,050.00	
Iron	4,410.00	ug/l	4.41	
Magnesium	7,420.00	ug/l	7.42	
Manganese	51.60	ug/l	0.05	
Molybdenum	33.60	ug/l	0.03	J
NG	400.00	ug/l	0.40	<
Nickel	189.00	ug/l	0.19	
Nitrate-N	2,390.00	mg/l	2,390.00	
Nitrite-N	5,840.00	mg/l	5,840.00	
o-Phosphate-P	368.00	mg/l	368.00	
Potassium	403,000.00	ug/l	403.00	
Sodium	72,680,000.00	ug/l	72,680.00	
Sulfate	274.00	mg/l	274.00	
Zinc	476.00	ug/l	0.48	
TIC	521.00	mg/l	521.00	
TOC	12,300.00	mg/l	12,300.00	
COD	32,700.00	mg/l	32,700.00	
Total Suspended Solids	364.00	mg/l	364.00	
Total Dissolved Solids	224,000.00	mg/l	224,000.00	
Normality as NaOH	3.20	n		
Density	1.19	g/ml		

J = Estimated Value; concentration is below limit of quantification

< = Concentration is below detection limit

Note: Off-gas production data was not collected for Run 19.

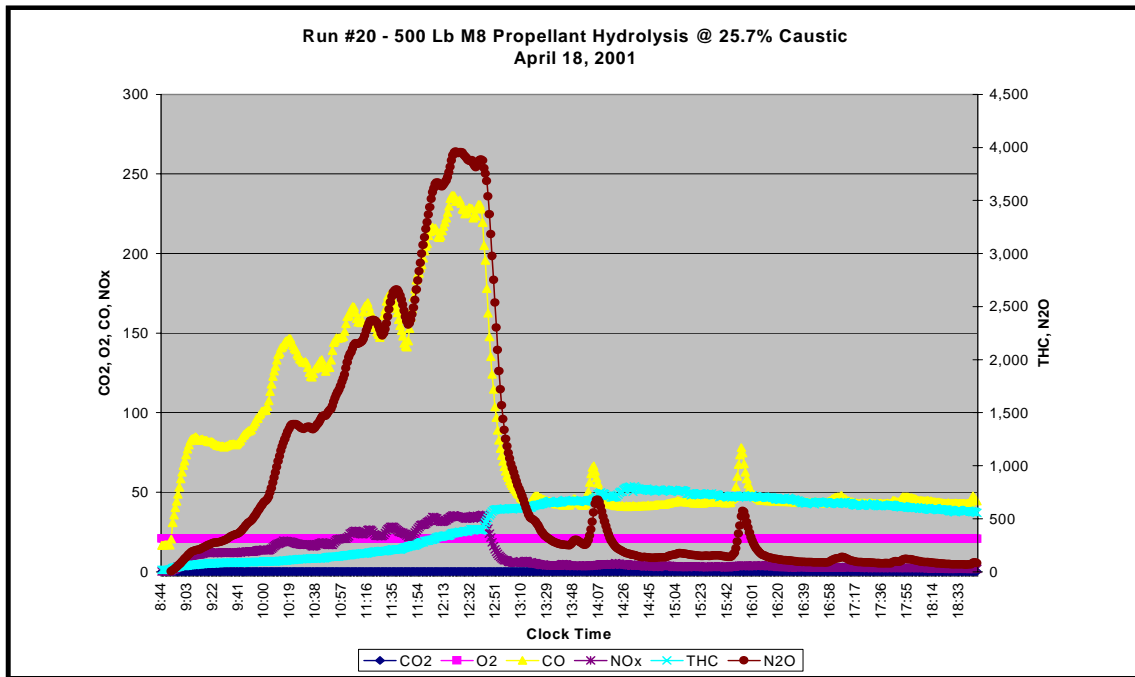


Figure 6-42. Run 20, Off-gas Production

Table 6-31. Run 20, M8 Propellant End of Run Hydrolysate Characterization

End of Run Hydrolysate Analysis				
Component	Concentration	Unit	ppm	Note
Acetate	4,320.00	mg/l	4,320.00	
Aluminum	265.00	ug/l	0.27	
Ammonia	74.50	mg/l	74.50	
Barium	78.20	ug/l	0.08	
Calcium	36,000.00	ug/l	36.00	
Chromium	148.00	ug/l	0.15	
Cobalt	48.00	ug/l	0.05	J
Copper	511.00	ug/l	0.51	
Cyanide (Sodium Cyanide)	133,000.00	ug/l	133.00	
Fluoride	291.00	mg/l	291.00	
Formate	1,380.00	mg/l	1,380.00	
Iron	1,800.00	ug/l	1.80	
Magnesium	18,200.00	ug/l	18.20	
Manganese	35.90	ug/l	0.04	
Molybdenum	33.60	ug/l	0.03	J
Nickel	216.00	ug/l	0.22	
Nitrate-N	675.00	mg/l	675.00	
Nitrite-N	1,580.00	mg/l	1,580.00	
o-Phosphate-P	100.00	mg/l	100.00	
Potassium	118,000.00	ug/l	118.00	
Sodium	34,550,000.00	ug/l	34,550.00	
Sulfate	74.50	mg/l	74.50	
Zinc	219.00	ug/l	0.22	
TIC	262.00	mg/l	262.00	
TOC	6,560.00	mg/l	6,560.00	
COD	14,900.00	mg/l	14,900.00	
Total Suspended Solids	348.00	mg/l	348.00	
Total Dissolved Solids	158,000.00	mg/l	158,000.00	
Normality as NaOH	2.60	n		
Density	1.14	g/ml		

J = Estimated Value; concentration is below limit of quantification



Table 6-32. Run 20, M8 Propellant Off-gas Characterization

Reactor Off Gas Analysis					
Component	During Energetic Addition	Note	During Reaction	Note	Unit
2-Butanone	439.00		642.00		ppbv
Acetaldehyde	7,780.00	D	8,180.00	D	ug/m <sup>3</sup>
Acetone	3,080.00		5,290.00		ppbv
Ammonia	425,000.00		1,830,000.00		ug/m <sup>3</sup>
Benzene	1,390.00		221.00		ppbv
Butanal	434.00		245.00		ug/m <sup>3</sup>
Carbon Dioxide	0.02		0.04		%
Carbon Monoxide	119.00		31.00		ppmv
Chloromethane	29.30		16.00		ppbv
Crotonaldehyde	39.90		18.90		ug/m <sup>3</sup>
Cyanide	0.07		0.05		mg/m <sup>3</sup>
Cyclohexanone	135.00		51.90		ug/m <sup>3</sup>
Decanal	13.80		5.33	J	ug/m <sup>3</sup>
Formaldehyde	336.00	B	204.00	B	ug/m <sup>3</sup>
Heptanal	24.80		8.73		ug/m <sup>3</sup>
Hexanal	69.90		38.90		ug/m <sup>3</sup>
Isopropyl Alcohol	24,968.10	J	129,625.60	J	ppbv
m-Tolualdehyde	348.00		263.00		ug/m <sup>3</sup>
NG	21,800.00				ug/m <sup>3</sup>
Nitrous Oxide	1,707.00		358.00		ppmv
Nonanal	35.30		6.99		ug/m <sup>3</sup>
NOx	18.10		2.90		ppmv
Octanal	15.00		8.09		ug/m <sup>3</sup>
Oxygen	21.20		21.20		%
Pentanal	81.50		16.00		ug/m <sup>3</sup>
Toluene	20.80		9.80		ppbv
Total Hydrocarbons	224.00		887.00		ppmv

J = Estimated Value; concentration is below limit of quantification.

D = Result obtained from analysis of a dilution or surrogate diluted below detection limit.

B = When applied to anions or organic analysis the qualifier indicates that the analyte was detected in the associated method/instrument blank.

### 6.5.2 Cotton Threads:

At the conclusion of each run, the cotton threads from the M8 propellant bundles were wrapped around the lower agitator and shaft. The agglomeration of threads was removed from the shaft before the start of the next run. Also, threads adhering to the perforated recycle pipe were scrapped-off and removed from the reactor. Figure 6-43 below shows the threads that were removed from the reactor.



Figure 6-43. Recovered Cotton Threads from Hydrolysis of M8 Sheet Propellant Bundles

The cotton threads can present a problem to valves and pumps, and need to be addressed in the design of the full-scale facility.

### 6.5.3 Discussion & Analysis:

1. The end of run liquid analyses indicates that the destruction rate efficiency achieved approximately 5 hours after cessation of the feeding of M8 propellant to the reactor ranged from 99.9828% to 100.00%. The end of run analyses for Runs 18 and 19 indicated destruction rate efficiencies for NG of 99.9600 and 99.9985 respectively, which is surprising since NG is readily destroyed by caustic.
2. The only "bad actor" detected in the hydrolysate is cyanide (possibly sodium cyanide) at a range of 133 to 378 ppm. However, as stated in paragraph 6.1.2 above, the hydrolysis of energetics will produce cyanide and that the subsequent treatment of said hydrolysate using SCOW technology adequately reduces the hazardous compound concentrations in the hydrolysate feeds well below levels of concern.
3. The airflow across of the reactor headspace was maintained at ~40 scfm during the test. The off gassing quickly fell-off after the addition of the propellant was completed (see Figures 6-41 and 6-42), indicating that the reaction was progressing to conclusion in line with the liquid analysis data. Unlike with M1 and M8 propellants, the THC level also dropped-off quickly after the addition of the M28 propellant was completed.
4. The heat released by the exothermic reaction easily controlled by the reactor jacket cooling system, and the hydrolysate was maintained at the 87°C set point without difficulty.
5. The operating level in the reactor was maintained just above the lower impeller representing a starting volume of ~700-gallons. Foaming was not experienced with M8 propellant.
6. Examination of the off-gas characterization for the three runs indicates that NG vapor was in the gas stream only during the addition phase of the process. NG has a measurable vapor pressure at ambient, therefore one would expect NG to come-off as part of the off-gassing stream. Use of a condenser to drop any entrained materials back into the reactor should be effective; however, the condenser should be designed for potential service with NG vapors and cleaned accordingly when the system is decommissioned from service.
7. Other bad actors in the gas stream included low levels of benzene and toluene.
8. The major constituent of the off gassing was ammonia, which dominated the characterization. Other constituents at significantly lower levels were acetaldehyde, acetone, isopropyl alcohol and nitrous oxide. These gasses can be effectively treated with a scrubber system, with the water from the scrubber then processed through the SCWO system as the final treatment step before release.

## 6.6 M28 Leaded Propellant and Composition B4 Mixture Test & Result (Run 21).

The objective of these tests is to clearly determine and define the optimum operating parameters for the M28 leaded propellant mixed with Composition B4 explosive (86/14 wt%) hydrolysis process to support the design and installation of the full-scale hydrolysis process at the Blue Grass Chemical Agent Disposal Facility for the destruction of propellant and explosives contained in the 115mm Rocket and Rocket Warhead, Chemical Agent GB & VX, M55 and M56.

The table below identifies the process operating parameters for Run 21.

Table 6-33. M28 Propellant (Leaded) and Composition B4 Explosive Mix Test Parameters

Operating Condition	Run 21
M8 Feed Rate, lbs/hr	
1 <sup>st</sup> hour	50
2 <sup>nd</sup> hour	100
3 <sup>rd</sup> hour	150
4 <sup>th</sup> hour	200
Caustic Soda Concentration, wt%	20
Caustic Soda Feed, gal	700
Reactor Operating Temperature, °C	87
Agitation Speed, RPM	80
Date Conducted	4/19/2001

The following chart presents the process operating and control parameters for the hydrolysis for M28 Leaded Propellant / Composition B4 Explosive mixture:

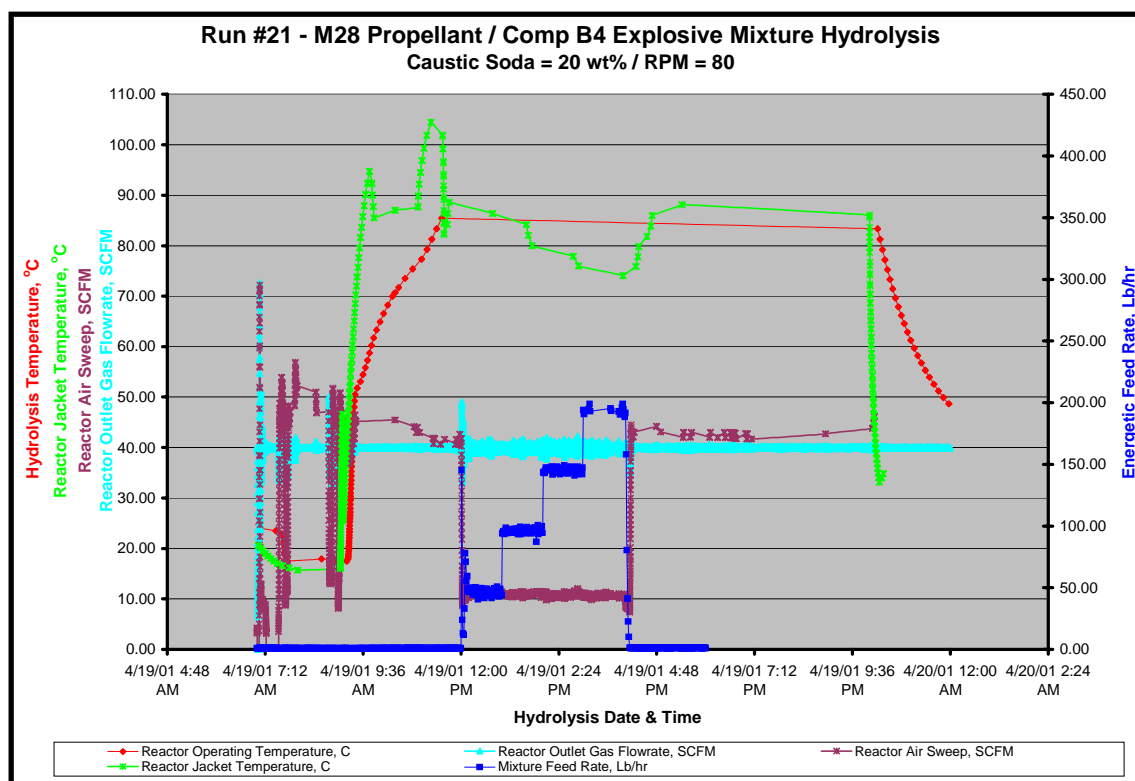


Figure 6-44. Run 21, Process Operating and Control Parameters

## 6.6.1 Tests Results:

The plots below represent the destruction of M28 leaded propellant and Composition B4 explosive mixture as a function of reactor residence time.

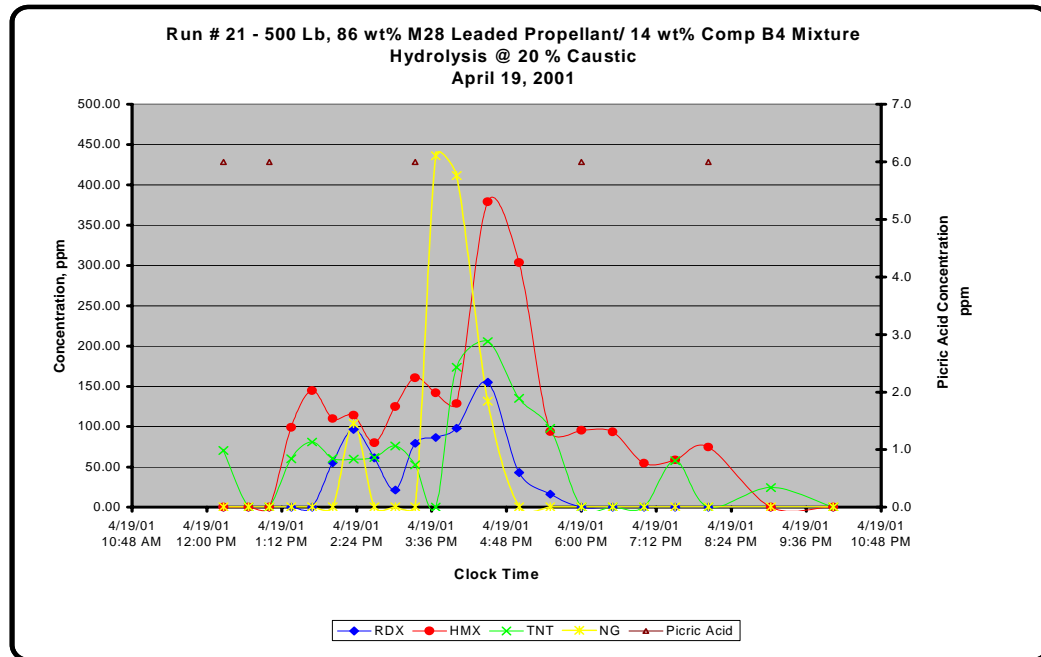


Figure 6-45. Run 21, Mid-Run M28 Propellant / Composition B4 Explosive Destruction Efficiency

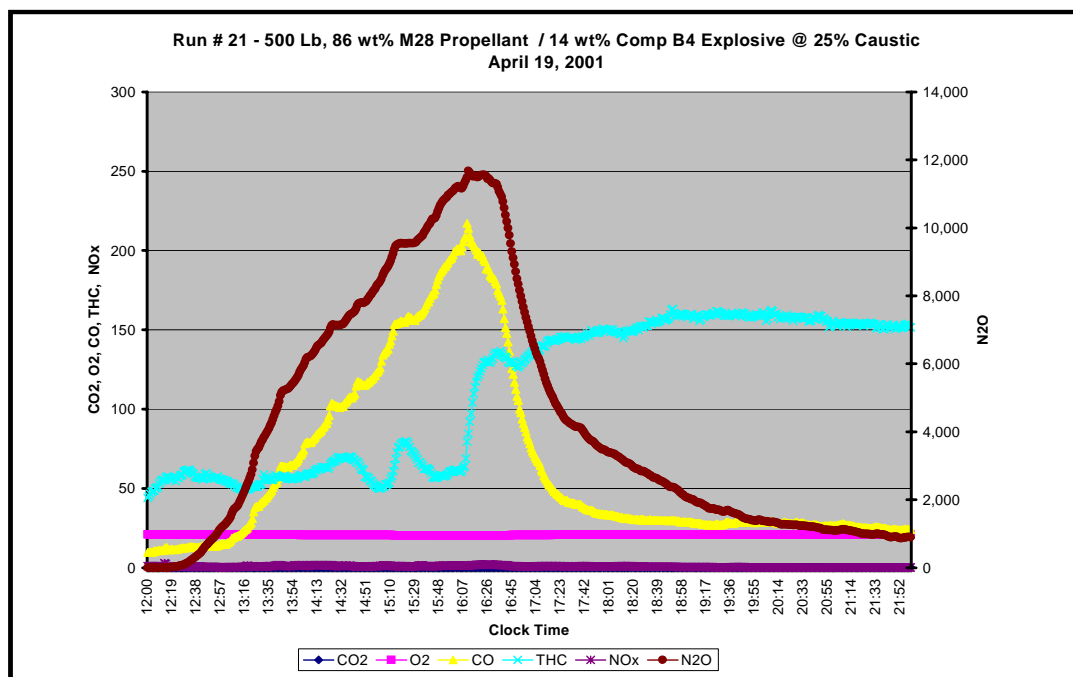


Figure 6-46. Run 21, Off-gas Production

Table 6-34. Run 21, M28 Propellant (Leaded) and Composition B4 Explosive End of Run Hydrolysate Characterization

End of Run Hydrolysate Analysis				
Component	Concentration	Unit	ppm	Note
Acetate	3,360.00	mg/l	3,360.00	
Aluminum	340.00	ug/l	0.34	
Ammonia	112.00	mg/l	112.00	
Barium	295.00	ug/l	0.295	
Calcium	35,900.00	ug/l	35.9	
Chromium	74.20	ug/l	0.0742	
Cobalt	22.30	ug/l	0.0223	J
Copper	297.00	ug/l	0.297	
Cyanide (Sodium Cyanide)	65,300.00	ug/l	65.3	
Di-n-butylphthalate	340.00	ug/l	0.34	J
Fluoride	230.00	mg/l	230.00	
Formate	1,240.00	mg/l	1,240.00	
HMX	24,180.00	ug/l	24.18	
Iron	1,110.00	ug/l	1.11	
Lead	79,300.00	ug/l	79.3	
Magnesium	9,740.00	ug/l	9.74	
Manganese	20.10	ug/l	0.0201	J
Molybdenum	36.70	ug/l	0.0367	J
Nitrate-N	472.00	mg/l	472.00	
Nitrite-N	1,310.00	mg/l	1,310.00	
Nitrocellulose	0.09	ug/l	0.00009	J
Phenol	390.00	ug/l	0.39	J
Potassium	4,540.00	ug/l	4.54	J
Sodium	25,030,000.00	ug/l	25030	
Sulfate	79.00	mg/l	79.00	
Zinc	119.00	ug/l	0.119	
TNT	29,790.00	ug/l	29.79	
TIC	140.50	mg/l	140.50	
TOC	4,547.25	mg/l	4,547.25	
COD	9,510.00	MG/L	9,510.00	
Total Dissolved Solids	66,500.00	mg/l	66,500.00	
Total Suspended Solids	330.00	mg/l	330.00	
Normality as NaOH	1.00	n		
Density	1.04	g/ml		

J = Estimated Value; concentration is below limit of quantification

Table 6-35. Run 21, M28 Propellant (Leaded) and Composition B4 Explosive Off-gas Characterization

Reactor Off Gas Analysis					
Component	During Energetic Addition	Note	During Reaction	Note	Unit
1,3,5-Trinitrobenzene	0.7250	MAX			ug/m <sup>3</sup>
1,3-Dinitrobenzene	0.8280	MAX			ug/m <sup>3</sup>
2,4,6-Trinitrotoluene	106.0000	MAX			ug/m <sup>3</sup>
2,4-Dinitrotoluene	14.9000	MAX			ug/m <sup>3</sup>
2,6-Dinitrotoluene	0.1860	MAX			ug/m <sup>3</sup>
2-Amino-4,6-Dinitrotoluene	0.2770	MAX			ug/m <sup>3</sup>
4-Amino-2,6-Dinitrotoluene	0.4610	MAX			ug/m <sup>3</sup>
Acetaldehyde	11.6000	B	2.4300	B	ug/m <sup>3</sup>
Acetone	3,030.0000		16.0000	U	ppbv
Ammonia	73,300.0000		303,000.0000		ug/m <sup>3</sup>
Bromomethane	78.7000		41.3000		ppbv
Butanal	0.3600		0.0306	J	ug/m <sup>3</sup>
Carbon Dioxide	0.0200		0.0100		%
Carbon Monoxide	0.0000		0.0000		ppmv
Chloroform	58.2000		16.0000	U	ppbv
Crotonaldehyde	0.1180	J	0.0683	J	ug/m <sup>3</sup>
Cyanide	0.0003		0.0007		ug/m <sup>3</sup>
Cyclohexanone	0.7020	J	0.0264	JB	ug/m <sup>3</sup>
Ethylenimine			12,839.5000	J	ppbv
Formaldehyde	17.8000	B	11.2000	B	ug/m <sup>3</sup>
Heptanal	0.1410	J	0.0048	U	ug/m <sup>3</sup>
Hexanal	1.4700		0.1740	J	ug/m <sup>3</sup>
Methyl Chloride	25.2000	B	24.0000	U	ppbv
m-Tolualdehyde	0.7750		0.0057	U	ug/m <sup>3</sup>
Nitrous Oxide	1,403.0000		276.0000		ppmv
Nonanal	0.1460	J	0.0461	J	ug/m <sup>3</sup>
NOx	0.0000		2.1000		ppmv
Octanal	0.0873	J	0.0258	J	ug/m <sup>3</sup>
Oxygen	21.2000		20.8000		%
Pentanal	1.5400	B	0.0469	JB	ug/m <sup>3</sup>
Tetryl	21.8000	MAX			ug/m <sup>3</sup>
Total Hydrocarbons	540.0000		1,326.0000		ppmv

J = Estimated Value; concentration is below limit of quantification.

MAX = Reported result was from a multi-fraction gas sampling train that contains both non-detected results and positive results.

U = Analyte was not detected.

D = Result obtained from analysis of a dilution or surrogate diluted below detection limit.

B = When applied to anions or organic analysis the qualifier indicates that the analyte was detected in the associated method/instrument blank

#### 6.6.2 Neutralization:

This mixture was disposed of as hazardous waste because of the lead content in the M28 propellant formulation (~2 % lead stearate). Therefore, the pH was adjusted to ~10 using concentrated sulfuric acid (98+%), introducing the acid through the new spray nozzle at a slow rate and keeping the hydrolysate temperature at less than 50°C. The procedure worked satisfactorily although NO<sub>x</sub> emissions were again generated during the neutralization reaction.

#### 6.6.3 Discussion & Analysis:

1. The end of run liquid analyses indicates that a 99.9172 % destruction rate efficiency was achieved approximately 6 hours after cessation of the feeding of M28 propellant / Composition B explosive mixture to the reactor – approximately 10 hours after the start of the run. HMX and RDX are detected in the hydrolysate, which was again surprising since both these energetics are readily destroyed by caustic. A low level of Picric acid was detected in the mid-run samples analysis as shown in Figure 6-45; however, Picric Acid was not detected in the end of run hydrolysate sample analysis.
2. The energetics loading for the M28 leaded propellant and Comp B explosive mixture is approximately 6.4 weight-percent resulting in a total solids (dissolved and suspended) loading at the end of the run approximately 7.7 weight-percent at 20 wt% caustic strength.
3. The only “bad actor” detected in the hydrolysate is cyanide (possibly sodium cyanide) at ~65 ppm and lead as Lead Hydroxide and/or Lead Picrate at ~79 ppm. As stated in paragraph 6.1.2 above, the hydrolysis of energetics will produce cyanide and that the subsequent treatment of said hydrolysate using SCOW technology will adequately reduce the hazardous compound concentrations in the hydrolysate feeds well below levels of concern. The lead is sourced from the lead stearate, which is 2% of the M28 propellant formulation. A low level of HMX and TNT (29.79 and 24.18 ppm respectively) remains at the end of run.
4. The airflow across of the reactor headspace was maintained at ~40 scfm during the test. The off-gassing quickly fell-off after the addition of the propellant was completed (see Figure 6-46), with the exception of THC<sub>s</sub>, indicating that the reaction was progressing to completion in line with the liquid analysis data. As previously stated, it is conjectured that the continued THC off-gassing was the result of dissolved gases and the continued reaction of the caustic solution with the by-products of the destruction of the NC chain (contributed by the M28 propellant).
5. The heat released by the exothermic reaction easily controlled by the reactor jacket cooling system, and the hydrolysate was maintained at the 87°C set point without difficulty.
6. The operating level in the reactor was maintained just above the lower impeller representing a starting volume of ~700-gallons. Foaming was not a problem with this mixture.
7. Examination of the off-gas characterization for the run indicates that some energetic materials (1,3,5-Trinitrobenzene, Dinitrotoluene, TNT and Tetryl) at low level were entrained in the gas stream during the addition phase of the process. TNT is to be expected since some dusting of the Composition B4 explosive will occur as the material fell into the reactor across the air stream. The Tetryl identification may have been residuals left in the traced Teflon gas sampling line back to the Instrumentation Van, or in the condenser system. The energetic materials disappeared once the addition was stopped. Use of a condenser to drop any entrained materials back into the reactor should be effective.



8. The major constituents of the off gassing were ammonia, acetone and nitrous oxide with ammonia dominating. These gasses can be effectively treated with a scrubber system, with the water from the scrubber then processed through the SCWO system as the final treatment step before release. The average concentration of CO<sub>2</sub>, O<sub>2</sub>, CO, THC, NO<sub>x</sub> and N<sub>2</sub>O in the off gas stream during energetic addition for this run was: 0.05%, 21.0%, 71 ppmvd, 86 ppmvd, 0.6 ppmvd, 5,385 ppmvd, respectively and during digestion the concentration was: 0.03%, 21.1%, 10 ppmvd, 211 ppmvd, 0.0 ppmvd, 895 ppmvd, respectively.
9. Lead Material Balance:

Lead Stearate comprised approximately 2% of the 430 pounds of M28 propellant used in the M28 propellant / Composition B explosive mixture test run, or approximately 8.6 pounds. The molecular weight (MW) of Lead Stearate is 774.15 and the MW of lead is 207.2. Therefore, the amount of Lead in the propellant is:

$$207.2 / 774.15 \text{ MW} \times 8.6 \text{ pounds} = \sim 2.302 \text{ pounds}$$

The Lead detected in the hydrolysate was 79.3 ppm, or equivalent to 79.3 mg/liter. The volume of the hydrolysate was approximately 800-gallons; therefore, the amount of Lead in the hydrolysate is:

$$79.3 \text{ mg/liter} \times 800\text{-gallons} \times 3.785 \text{ liters/gallon} = \sim 240120 \text{ mg or } \sim 0.53 \text{ pounds}$$

Consequently, ~ 78% of the Lead is unaccounted for, which is mostly likely to be found in the un-dissolved solids content of the hydrolysate in the form of lead hydroxide.

A second analysis performed on the hydrolysate to confirm the Lead level showed a 78.1 ppm concentration.

## 7.0 M1 PROPELLING CHARGE CONFIGURATION ISSUE -- CLOTH BAGS

The M1 propellant in the 105mm cartridges is configured in cloth bags for the purposes of zoning the round. The testing conducted under this program used loose M1 propellant since the M1 loaded propellant bags were not available in the stockpile. However, LANL was asked to conduct tests with the cloth bag material (spun viscose rayon, resin impregnated class 1, spec MIL-C-43157)) to determine if the cloth bag could be destroyed in the caustic solutions being used to hydrolyze the propellant; thereby saving downloading time (removal of the M1 propellant from the bag).



Figure 7-1. M1 Propellant Grains

### 7.1 Experiments with the Rayon Cloth.

Two sets of four swatches of rayon cloth were exposed to 6-, 12- and 20-weight percent NaOH at 93°C for 340 min. The swatches were photographed before and after the exposure to base. Mass loss of the swatches could not be determined due to a large amount of NaOH crystals adhering to the fibers when dried. Repeated rinsing of the swatches with water did not appear to improve the situation

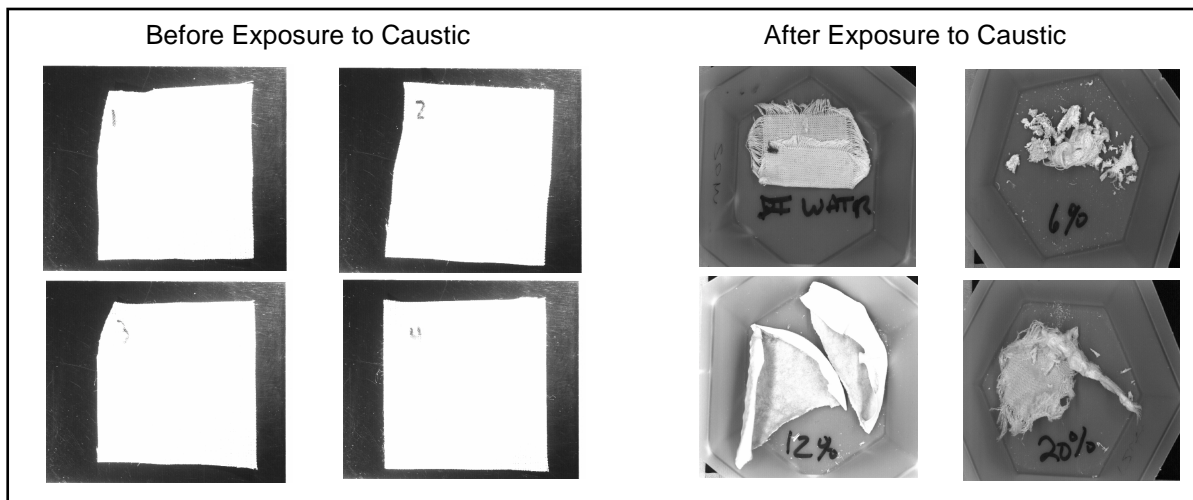


Figure 7-2. Rayon Cloth Samples

### 7.2 Recommendation.

The handling of the M1 propellant charges may present a problem. The rayon bag containing the M1 propellant was not digested during the testing conducted at LANL; however, it is conjectured that processing the rayon bag in caustic through an in-line homogenizer would most likely break-up the bag into small pieces that could then be effectively digested by the caustic solution. Bottom Line: The handling of the M1 propellant charges as a separate campaign should also address the handling of the rayon bag.

## **8.0 HYDROLYSATE PREPARATION FOR USE IN SCWO TEST AND EVALUATION PROGRAM**

The PM decided late in the program to use the hydrolysates produced during the equipment commissioning and the test and evaluation program to support the continued testing of the Foster-Wheeler SCWO reactor at DPG. A defined hydrolysate specification did not exist. After much discussion, Foster-Wheeler provided the following recipe for hydrolysate: 9.2% energetic solids loading reacted with a 20% NaOH solution.

Since the hydrolysates were already produced, it was decided to execute the recipes in the laboratory at Holston AAP using Composition B explosive and M1 propellant individually so as to convert the proposed explosive loading into an actually measured solids loading. The results of that work are summarized below:

### **8.1 Composition B Explosive Hydrolysate.**

#### **8.1.1 Laboratory Procedure:**

1,000-grams of 20.19% caustic solution was prepared and charged to a standard (Holston AAP) 3-liter still. The caustic solution was heated to 87°C and 101.33-grams of Composition B was incrementally charged to the caustic solution over an approximate 5-minute period (representing the above stated recipe). The reaction mixture was held at 87°C ± 3°C for 6-hours. The hydrolysate was then cooled to 50°C, drained from the still into a Nalgene storage bottle and was allowed to sit overnight. The hydrolysate was filtered at 26°C through a coarse porosity glass crucible (approximate 20-micron pore size). Samples of the hydrolysate were taken to determine dissolved solids. 500-grams of the filtered hydrolysate was neutralized using 173.27-grams of 60.2% nitric acid. The neutralized hydrolysate was tested to determine suspended and dissolved solids.

#### **8.1.2 Laboratory Results – Solids Loading:**

##### **Composition B Hydrolysate:**

- % NaOH 18.78% (average of 2 readings)
- Suspended Solids 0.25%
- Dissolved Solids 27.00% (average of three tests at 27.03%, 27.02%, and 26.96%)
- Total Solids 27.25%

##### **Neutralized Composition B Hydrolysate:**

- pH 8.3
- Suspended Solids 0.02%
- Dissolved Solids 29.17% (average of three tests at 29.20%, 29.18%, and 29.13%)
- Total Solids 29.19%

### **8.2 M1 Propellant Hydrolysate.**

#### **8.2.1 Laboratory Procedure:**

1,000-grams of 20.19% caustic solution was prepared and charged to a standard (Holston AAP) 3-liter still. The caustic solution was heated to 87°C and 101.33-grams of M1 propellant was incrementally charged to the caustic solution over an approximate 5-minute period (representing the above stated recipe). The reaction mixture was held at 87°C ± 3°C for 6-hours. The hydrolysate was then cooled to 50°C, drained from the still into a Nalgene storage bottle and was allowed to sit overnight. The hydrolysate was filtered at 26°C through a coarse porosity glass crucible (approximate 20 micron pore size). Samples of the hydrolysate were taken to determine dissolved solids. 500-grams of the filtered hydrolysate was neutralized using 176.69-grams 60.2% nitric acid. The solution was slightly acidic with an approximate pH = 6. Therefore, 9.98-grams of 20.19% NaOH was added to the mixture to increase the desired pH within the targeted range of 7 –10. The neutralized hydrolysate was tested to determine suspended and dissolved solids.

### 8.2.2 Laboratory Results – Solids Loading:

#### M1 Hydrolysate:

- % NaOH 15.21% (average of 2 readings)
- Suspended Solids 0.29%
- Dissolved Solids 27.12% (average of three tests at 27.11%, 27.05%, and 27.19%)
- Total Solids 27.41%

#### Neutralized M1 Hydrolysate:

- pH 9.8
- Suspended Solids 0.07%
- Dissolved Solids 30.73% (average of three tests at 31.04%, 30.50%, and 30.65%)
- Total Solids 30.80%

## 8.3 Particle Size.

Using the design documents for the Pueblo Chemical Agent Disposal Facility, General Atomics proposes to produce energetics hydrolysate at a rate of 400-700 lbs/hr, and cites a total of 5,000 gallons of storage tank capacity in the Energetics Rotary Hydrolyzer and the Hydrapulper before the hydrolysate is fed to SCWO. The hydrolysates will typically be held for 2.5 - 4.5 days before the hydrolysate is fed to the SCWO. Parsons cites similar flow rates with a 1,600-gallon energetics hydrolysate holding tank, so the energetics hydrolysate will typically be held for 1.5 days before the hydrolysate is fed to the 16,000-gallon ICB feed tank, where it is neutralized and diluted. Therefore, solids that may form after neutralization and storage on the order of weeks should not be relevant to the proposed processes.

## 8.4 Hydrolysate Prepared for DGP.

The hydrolysates stored in the tank farm were neutralized and shipped via tanker to DPG:

#### Propellant Hydrolysate:

- Shipping Date: 8/16/01
- Trailer No.: 4231
- Weight (lbs): 40,680 lbs
- pH: 9.8
- % Solids (Total): 20.97% (average of six samples within a range of 20.85% - 21.05%)
- Batches: P-N-1, P-N-2, P-N-3, P-N-4
- HNO<sub>3</sub> (65%) Added: ~665 gallons

- Shipping Date: 8/23/01
- Trailer No.: 4853
- Weight (lbs): 42,660 lbs
- pH: 9.4
- % Solids (Total): 20.76% (Average of 6 Analyses)
- Batches: P-N-4, P-N-5, P-N-6, P-N-7
- HNO<sub>3</sub> (65%) Added: ~715 gallons

#### Explosive Hydrolysate:

- Shipping Date: 8/17/01
- Trailer No.: 71404
- Weight (lbs): 43,840 lbs
- pH: 9.7
- % Solids (Total): 20.35% (average of six samples within a range of 20.28% -20.42%)
- Batches: E-N-1, E-N-2, E-N-3, E-N-4
- HNO<sub>3</sub> (65%) Added: ~767 gallons

- Shipping Date: 8/22/01
- Trailer No.: 11465
- Weight (lbs): 44,960 lbs
- pH: 9.8
- % Solids (Total): 21.13% (Average of 6 Analyses)
- Batches: E-N-5, E-N-6, E-N-7, E-N-8
- HNO<sub>3</sub> (65%) Added: ~764 gallons

The percent solids values provided are the total solids in the hydrolysate (suspended and dissolved).

#### 8.4.1 Filtering of the M8 Propellant Hydrolysate:

Before neutralizing the M8 propellant hydrolysate, which was stored separately from the M1 and M28 propellant hydrolysates, the cotton threads had to be filtered from the solution. This was performed using two in-line screen filters called Candle Filters. The dimensions of the overall filter were 44-inches long by 6-inches in diameter. The internal filter cartridge was 34-inches long by 3-inches in diameter. The internal filter essentially consisted of a 3-inch open pipe wrapped with a mesh screen connected to the pipe by stainless steel wire. The mesh size of the screen is 3/32 square inch mesh with 1/16 square inch actual opening size. The Figure 8-1 shows the threads that were removed from the hydrolysate.



Figure 8-1. In-line Screen Filters and Recovered Cotton Threads from M8 Propellant Sheet Bundles

Based on the amount of cotton threads recovered from the in-line screen filters, it would appear that the bulk of the cotton threads remained in the reactor (wrapped around the impeller blades) and were recovered from the reactor at the end of each run (see Figure 6-43).

#### 8.4.2 Neutralizing with Nitric Acid:

Foster-Wheeler, the contractor for the SCWO system that required the hydrolysates, requested that the hydrolysate be neutralized with  $\text{HNO}_3$  as opposed to  $\text{H}_2\text{SO}_4$ . The neutralization proceeded without problems, the  $\text{HNO}_3$  proved to be much “friendlier” than the  $\text{H}_2\text{SO}_4$  from a processing standpoint. The  $\text{H}_2\text{SO}_4$  (98%+) is hygroscopic, so much so that there is a significant heat release during dilution alone. The enthalpy of dilution, coupled with the enthalpy of neutralization is much, much greater than that for  $\text{HNO}_3$  (50-60% strength). This accounts for the significant temperature rise that was experienced during Run 1 neutralization using strong  $\text{H}_2\text{SO}_4$ .

## **9.0 EQUIPMENT / HARDWARE “LESSONS LEARNED”**

### **9.1 Acrison Loss-in-Weight Feeder.**

The performance of the Acrison loss-in-weight feeder was poor throughout the program. The major problem was during start-up of the feeder. The drive motor for the conditioning and feed auger repeatedly stopped because of a current overload at start-up. This problem is attributed to the design of the conditioning auger, which was modified before starting the M28 propellant tests. The modification appeared to solve the problem. Other problems included:

- The control system was not properly interfaced/designed for remote operation (i.e., operators were required to enter the processing building to reset frequent alarms).
- The control system would occasionally report an incorrect total mass greater than that loaded into the feed hopper (several percent higher).

The technical support from the vendor was also unsatisfactory. The vendor was reluctant to visit the work site to trouble shoot the problem, and when the problem was traced to the conditioning auger configuration, the modifications were executed by RONA. The aggressive program schedule contributed to problem of working with the vendor to resolve the feeder performance problems.

### **9.2 Recirculation Line.**

The performance of the recirculation line was marginal. The pump had difficulty pulling suction sufficient to initiate the recirculation flow at start-up. In addition, the direction of the flow, pulling from the top of the reactor and pumping into the bottom of the reactor, contributed to aeration. While this ensured that the discharge valve would not clog, it also impacted the performance of the mass flow meter, which is sensitive to aeration of the flow. This also impacted the performance of the liquid sampling system at times.

A preferred approach would be to design the reactor recirculation loop for bi-directional flows, (e.g., adding a second inlet/outlet port at the bottom of the reactor or a side port with the appropriate piping). The NRC and potential facility contractors asked, “Do we need the recirculation feature in the full-scale facility?” The recommendation is to include the recirculation loop for the flexibility it provides. The hardware and labor cost associated with installing this loop is insignificant compared to the total cost of the chemical weapons demilitarization facility.

### **9.3 Mass Flow Meter.**

The performance of the mass flow meter was unsatisfactory because the meter was installed before the suction inlet to the recirculation pump, cause aeration to occur, especially during the energetics addition stage of the process. Reinstalling the mass flow meter on the discharge side of the pump will solve the problem. There was not sufficient time in the schedule to reinstall the mass flow meter. From a facility standpoint, the mass flow meter is not essential to safe hydrolysis of energetics.

### **9.4 Agitator Shaft Seal.**

The use of water-seals for the reactor agitator, a requirement of RONA and the Holston AAP Safety Office, was proven to be a wrong choice. The recommended shaft seal is either a packing gland or a dynamic water-flushed graphite seal; both seals are readily available from equipment vendors as standard hardware. The potential for overflowing the reactor is probably a more serious concern than contamination of a seal.

### **9.5 Reactor Overflow Line.**

The over flow line from the reactor to the dump tank was installed at the wrong elevation. The overflow line should have been installed ~12-inches below the top of the reactor. This mistake caused the overflow line, a major fail-safe design element, to work improperly.

### **9.6 Sizing.**

The flow meters and valves for the acid and water lines were over sized by a considerable margin, which affected the ability to accurately measure flows at low rates. This was mostly of concern when introducing acid during the neutralization process, or trying to adjust pH of the hydrolysate by introducing additional caustic or water.

### **9.7 Commissioning Time Frame.**

Because of the aggressive schedule, the commissioning of the system was accelerated, resulting in problems later during the test program. Many changes that should have been made to improve the performance of the system had to be delayed or postponed indefinitely. However, the contractor as a necessary condition of operation to meet the schedule requirements accepted this.

### **9.8 Electrical / Instrumentation / Control.**

The following comment are germane to this program and should not be an issue for the implementation of a full-scale chemical weapons demilitarization facility:

- Designs for control systems are often done without consideration of maintenance, checkout, and startup activities. Provisions for devices to be electrically disconnected without shutting down an entire system are a requirement (e.g., fuses).
- PLC programmer not able to provide adequate support for the project time scale, largely because of time pressures but also because of lack of experience for energetic chemical processes. Required significant on-site support (expensive, time consuming).
- Rigidly structured, schedule driven programs do not allow flexibility for unknown situations and for routine abnormalities; an accepted cost of doing business for this program.
- Manual control of all field devices is a necessity for pilot plants as well as production facilities within the constraints of safety interlocks.

### **9.9 Positive Lessons Learned (Things That Worked Well!).**

- Redundant process monitoring equipment turned out to be very useful; e.g., measuring power consumption of recirculation pump, which allowed verification that hydrolysate was flowing in the recirculation line even when the mass flow meter displayed zero flow.
- Foaming can be effectively controlled using the level within the tank relative to the vortex formed by the agitator impeller, and the supplemental water spray (once the spray nozzle had been sized correctly).
- Modified Computer / PLC interface performed much better than might otherwise have been expected, and was worth the development effort.
- The reactor level sensor can detect foam.
- Manual overrides (e.g., temperature controls, valve controls) were vital to operating the hydrolysis system during the test program.
- PCS-7 style process monitoring (trends) can be exploited to learn more about the process.
- TRC feedbacks on reaction progress (the real time off-gas analyses) were a tremendous aid in tracking the progress of the reaction, especially during the commissioning runs.
- Modifying the liquid sampling system software for full programming of the sampling interval for the final few test runs simplified the duties of the control room operators.



## 10.0 CONCLUSIONS

The base hydrolysis, whether as individual energetic compositions or mixtures of energetics, is a proven process for the effective destruction of energetics recovered from the demilitarization of chemical weapons. The process is extremely robust, and provides a great deal of flexibility in terms of process rates, equipment selections and scaling.

The following responses are provided regarding the concerns cited by the NCR based on the bench-scale work performed by LANL and the pilot scale testing conducted at Holston AAP:

### 1. Destruction Rate Efficiency (DRE).

DREs ranged from a low of 99.7539 (Tetrytol) to 100.00% for all energetics processed. The tests performed on the hydrolysate show it to be safe to handle with the only intrinsic hazard being the high pH (13-14) of the final solution.

Several of the end of run analyses identified the presence of NG, HMX and RDX. This was surprising since these energetics are readily destroyed by caustic and/or had no clear source. It is possible that these unreacted materials may have been introduced from residuals in the sampling valve in the recycle line when the sample was drawn, biasing the results.

### 2. Optimum Processing Conditions.

Based on the energetics hydrolysis system testing performed at Holston AAP, it is concluded that the optimum processing conditions are: 20 weigh-percent sodium hydroxide, 87°C hydrolysate processing temperature, and 70-80 rpm agitator speed, and maximum 9-hour reactor resident time for propellants, explosives and mixtures. Using 25 weight-percent caustic had negligible effect on the reaction rate. Testing was performed with feed rates of 50-, 100-, 150- and 200-pounds per hour without difficulty. A feed rate of 500-pounds per hour was achieved during the commissioning trials with Composition B. Therefore, the hydrolysis process can safely process the energetic feed rates proposed for the Pueblo and Blue Grass Chemical Agent Disposal Facilities.

### 3. Characterization of Gas Generation.

The LANL bench-scale testing showed that the amount of gas produced during the hydrolysis of propellants was much less than that produced during the hydrolysis of Composition B4 or Tetrytol. Although all four major gas products ( $N_2$ ,  $N_2O$ ,  $NH_3$  and  $NO$ ) were detected, the quantity was minimal. A comparison between the rates at which gas is generated for the different energetics is depicted below:



The results of the full-scale testing conducted with the hydrolysis system correlates well with the LANL findings regarding gas generation and major gas products. Low levels of energetic materials (1,3,5-Trinitrobenzene, DNT, TNT, RDX, HMX, Tetryl, NG) are entrained in the off-gas stream during addition; however the energetics disappeared once the addition is completed. A condenser on top of the reactor and a water-mister will knock these materials back into the reactor. Cyanide, Benzene, Ammonia, Toluene, Xylenes, etc. that were detected in the off-gas stream can be effectively treated by a properly designed scrubber system.

#### **4. Energetic Residue.**

At the end of the test program, RONA personnel conducted a thorough inspection of the inside of the reactor to see if there are energetic materials coating the reactor. The following was observed:

- A crust like layer of energetic material was observed on the top of the reactor. This is attributed to commissioning Run 2 conducted at full capacity where foaming occurred. Most likely the foam carried with it energetic materials that coated inside the reactor.
- Upon inspection of the wall of the reactor within the working level, an insignificant layer of coating was observed and analyzed to determine its constituents. The result of this analysis is shown below: (awaiting results from RONA)

#### **5. Stack Off Gas Monitoring.**

The off gas leaving the scrubber in the stack was not monitored since this program was a pilot program and RONA was exempted from such monitoring. However, RONA did conduct visual monitoring and observed no unusual color or smell to the stack gas in the surrounding area.

#### **6. Rate of Reaction.**

LANL estimated the rates of reactions for the energetics in 12- and 20-weight percent caustic strength (see LANL Final Report). However, the particle sizes were not normalized; consequently, an absolute comparison cannot be made as to which material reacts the fastest and which material reacts the slowest. Based on work to date by LANL and with the pilot plant system, the slowest reacting energetics were the M1 and M8 propellants. The NC takes a long time to digest to its final by-products, as indicated by the sustained period of off gassing that occurred during the reaction. The Composition B/B4 and Tetrytol reacted very quickly with the reaction completed within 5-hours after the addition of energetics ceased. The M28 propellant reacted very quickly, more quickly than one would expect given its NC content. Some of this is attributed to the large percentage of NG, but another important element was the particle size of the surrogate M28 propellant (very small).

#### **7. Formation of Picric Acid.**

The formation of Picric Acid as a by-product of energetics hydrolysis is not a problem. Picric Acid was only detected at very low levels in the mid-run analyses for Tetrytol and was detected at a much lower level in the end of run analyses. This conclusion is supported by the bench-scale work performed by LANL that showed no Picric Acid present in the hydrolysate.

#### **8. Lead Material Balance (M28 Propellant).**

Approximately 22% of the lead introduced to the hydrolysis reaction as lead stearate in M28 propellant was accounted for in the hydrolysate (reported as ~79 ppm total Lead). The remaining lead is most likely in the suspended solids too low to account for the remaining (as lead hydroxide and/or lead picrate) analyzed at ~330 ppm. Further testing would be required to confirm this hypothesis.

## **9. Simultaneous Processing of Mixtures (Explosives and Propellants).**

A 500-pound mixture of M28 propellant (containing lead stearate) and Composition B explosive (86/14 weight-percentage ratio) was successfully process in the energetics hydrolysis system at Holston AAP. No problems were encountered. The DRE achieved was 99.999+ percent. The end of run analysis of the hydrolysate and the off-gas characterization tracked nicely with the analyses and characterizations obtained for the individual components. Processing this mixture, the most likely mixture to be encountered in the demilitarization of the chemical weapons, can be safely accomplished in the proposed Pueblo and Blue Grass Chemical Agent Disposal Facilities. This conclusion is supported by the bench-scale work performed by LANL.

## **10. Particle Size Reduction.**

The reactions involved in the hydrolysis of energetics are mass transfer limited, governed by the particle size of the materials present to the caustic solution (the smaller the particle size, the faster the rate of reaction). However, the explosives are all TNT based, and the TNT matrix quickly collapses at the operating temperature used during the test runs (87°C). Once the TNT melts (as well as reacts with the caustic), the particle size is controlled by material used in the formulation (RDX, HMX, and/or Tetryl). Particles sizes ranging from “fines” to chunks of Tetrytol approximately 1.25-inches in length and diameter were processed without difficulty. The propellant, specifically the M1 and M8, processed quickly and will not require any reduction. The M28 propellant is the only question mark -- how large will the particle sizes be when the M28 propellant is extracted form the rocket motor? The M28 propellant used in the testing was very small and not necessarily representative of the actual M28 propellant that will be seen by the production process. However, no problems are anticipated in processing ground M28 (grinding M28 propellant is being investigate at this time with Ecologic).

## **11. Cotton Threads and Rayon Bags.**

The handling cotton threads used to tie the M8 sheet propellant bundles must be addressed in the design of the energetic hydrolysis system. The rayon bags used to contain the M1 propellant should be emptied and then disposed of separately.

## **12. Solubility of Energetics.**

The aqueous concentrations of energetics is very low, even at high temperatures:

- RDX @ 90°C is ~300 ppm
- HMX @ 90°C is ~150 ppm
- TNT @ 60°C is ~675 ppm

Therefore, solubility is not a problem in designing the hydrolysis system for the proposed Pueblo and Blue Grass Chemical Agent Disposal Facilities

## **13. Heats of Reaction.**

LANL has provided an estimation of the heats of reactions for the various energetic compositions. These values can be used to calculate heat loading at various processing rate to assist in sizing the heat exchanger for the reactor jacket. However, developing heats of reaction for the by-products of the reaction represents a huge undertaking, and may not lend much value to the program.

#### **14. Foaming.**

Foaming was primarily controlled by adjusting the operating level of the hydrolysate within the reactor relative to the location of the vortex generated by the agitator blades. So long as a clearly defined vortex was maintained in the reactor, any foam formed during the hydrolysis would be quickly drawn below the surface and dissipated. Foaming was not experienced when processing propellants or the M28 propellant/Composition B explosive mixture.

#### **15. Tetryl Explosive.**

Tetryl explosive alone was not tested in the energetics hydrolysis system. Tetryl was not readily available from the inventory in the quantities required to test the system, and Tetryl presents handling problems (dusting) and is a health hazard. Furthermore, Tetryl is present in Tetrytol at 70+ percent. The testing with Tetrytol did not disclose any problems whatsoever processing the Tetryl constituent.

#### **16. Thermal Runaway.**

Based on work performed by LANL, the thermal runaway temperature for the propellants and explosives tested is 130°C or above for all base concentrations between (12 – 35 weight percent NaOH).

#### **17. Equipment Performance.**

The energetics hydrolysis reactor and supporting equipment performed acceptably during the test runs. While there were improvement identified that would have improved the operations during the test program, the aggressive schedule prevented most of these improvement from being implemented. However, for the most part the improvements were directed at improved data collection and experimentation, as opposed to correcting operating deficiencies, of which the only major problem was the loss-in-weight feeder that is not applicable to the proposed Pueblo and Blue Grass Chemical Agent Disposal Facilities. Of note, the system at Holston AAP was based on a 2000-gallon reactor. A smaller reactor, in the order of 500- to 1000-gallons volume, would be better suited for the proposed throughputs cited for the two facilities, based on the operational scenario chosen (batch versus semi-continuous operation).

#### **18. Equipment/System Maintenance.**

Over 300-hours of operating time were placed on the energetic hydrolysis system during the execution of the program. No major equipment failures were encountered during operations. The equipment used in the system is standard chemical processing hardware with extremely high degrees of reliability.

At the conclusion of the test runs, a Pfaudler representative visited Holston AAP to assess wear of the Glasteel® liner. Based on the manufacturing cards for the reactor, between 1-2 mils of glass was lost during the test runs on the bottom head of the vessel and about 3-feet up the side walls of the vessel, and similarly on the baffle, which is not very much. The glass was dull and rough to the touch, indicating that the "fire polish" was lost on this portion of the vessel. Loss of the polish indicates that the materials being processed are causing wear, but it in and of itself, is not going to accelerate wear. The only concern voiced by the Pfaudler, Inc. representative is that the roughen surface will tend to accumulate material and take more effort to clean, and the material accumulating on the Glasteel® will cause the continue corrosion. Pfaudler indicated that the usual approach by the user is to map the areas where some wear is occurring and monitor said area. When the Glasteel® thickness enters the 30-mil thickness is when closure scrutiny is required -- we are not close to that at this time. Bottom Line: The wear to date was not serious in the sense that the reactor was being compromised near term.

The Hastelloy C-276 agitator exhibited no wear whatsoever.



## APPENDIX A

### Acronyms and Abbreviations

ACWA	Assembled Chemical Weapons Assessment
AAD	Army Ammunition Depot
AAP	Army Ammunition Plant
ARDEC	Armament Research, Development and Engineering Center
°C	degrees Centigrade
CW	Chemical Weapons
DPG	Dugway Proving Ground
DRE	Destruction Rate Efficiency
EST	Engineering Scale Test
EDP	Engineering Design Package
°F	degrees Fahrenheit
ft <sup>2</sup>	square foot
FMEA	Failure Mode and Effects Analysis
FY	Fiscal Year
gal	gallon
g/l	grams per liter
HDPE	High Density Polyethylene
HSAAP	Holston Army Ammunition Plant
kJ/g	Kilo-joules per gram
LANL	Los Alamos National Laboratory
lbs/hr	pounds per hour
mg/l	milligrams per liter
ml	milliliter
NLT	No Later Than
NRC	National Research Council
NSWC	Naval Surface Weapons Center
PM	Program Manager
ppbv	parts per billion by volume
ppm	parts per million
ppmvd	parts per million volume density
RONA	Royal Ordnance North America
rpm	revolutions per minute
scc/g	standard cubic centimeter per gram
scfm	standard cubic feet per minute
SCWO	Super Critical Water Oxidation
SET	Solvated Electron Technology
SOP	Standard operating Procedure
TACOM	Tank-Automotive and Armaments Command
TOC	Total Organic Compounds
TPR	Test Plan Requirements
ug/l	micrograms per liter
ug/m <sup>3</sup>	micrograms per cubic meter
VOC	Volatile Organic Solvents
wt%	weight percent

## **APPENDIX B**

### **NRC Concerns**

## **Characterization & Optimization Responding to the NRC Concerns**

The following issues/concerns identified by the National Research Council (NRC) will be addressed during the Phase I design activities, and the test programs conducted during the Phase II and optional Phase III efforts.

### **1. Determine the particle size reduction of the energetic that must be achieved for proper operation:**

There are two issues associated with particle size:

- The particle size of the incoming energetics and its impact on the hydrolysis process
- The resulting particle size of the salts from the hydrolysis

Particle size of incoming energetics: As part of Phase 1, a bench-scale study will be initiated to determine the effect particle size has on the hydrolysis process. In addition, methodology will be investigated for handling the incoming energetics including the particle size of the energetics and final recommendations presented to PM ACWA. However, it may be difficult to reduce the size of energetic before introduction to the hydrolysis reactor by size reduction equipment because of safety considerations. Since reaction of the energetic with caustic will happen regardless of the particle size of the energetics, this office feels that it would be better to increase the alkaline concentration to assure completion of the hydrolysis. However, some work performed by Los Alamos National Laboratory (LANL) has indicated that there is a limit to this approach. If this approach is unsuccessful, then studies aimed at increasing the residence time or increasing the rate of agitation will be conducted.

Resulting particle size of the salts from the hydrolysis: Phase II will investigate optimizing particle size of the finished product through control of the reactor operating parameters. A determination will be made if some type of re-sizing equipment, such as a homogenizer, will be needed for the finished product (hydrolysate) stream. In this case, a homogenizer may be used in a reactor recycle loop as a means to simultaneously reduce the particle size of residual unreacted energetics and to increase the effective residence time.

### **2. Determine the solubility of energetic in specific alkaline solutions:**

Naval Surface Weapons Center (NSWC) and Los Alamos National Laboratory (LANL) will collaborate on solubility studies based on their extensive experience with the hydrolysis process chemistry. LANL will also review the present database on hydrolysis of energetic materials. Based on the LANL work, the solubility of these materials in alkaline solutions and an approach to prevent emulsification of the energetics will be developed and verified during the Phase II effort. Samples will be taken from the pilot plant during the trial runs of Phase II to determine solubility levels, and to assure that the method of hydrolysis chosen has resolved this problem.

### **3. Establish the process design of the unit operations and identification of the processing parameters:**

During Phase II, trial runs will be conducted at different residence times, feed rates, temperatures, pH, and agitation levels to optimize the hydrolysis process parameters and produce an acceptable final product. Samples of the hydrolysate will be collected to identify the products as a function of these optimized process parameters. During these runs, the following processing parameters will be refined:

- Determine the critical temperature for conducting the hydrolysis reaction based on efficiency (time of reaction, residence time, completeness of reaction and products produced). Operating temperature should not exceed 150°C for any energetic to avoid detonation.
- Quantify the amount of heat that must be absorbed during the exothermic reaction. Operating at a 20% caustic strength is being considered to ensure completeness of reaction and reduce the



residence time within the reactor. However, this will also put an additional demand on the cooling system for the reactor jacket.

- Determine the optimum rate for the addition of the energetics to the reactor. This is considered a critical parameter since it will determine the amount of heat liberated by the exothermic reactions as a function of time, and will also impact the thoroughness of the reaction, the demands on the agitation, and the possible formation of undesirable products.
- Establish the effective working volume for the reactor. If foaming occurs, or if there are volatiles, we need to ensure that the liquid/solid level in the reactor is not so high as to promote volatiles/solids from being driven off and into the air handling system.
- If required, determine the actions that must be taken to address the foaming associated with wax containing formulations; e.g., use of surfactants will be investigated to keep the wax fully dispersed in the hydrolysate mix.
- From a safety standpoint, develop a contingency plan to respond to a sudden shut down of the system. The concern is that the shutdown occurs during the early phase of the hydrolysis reaction when heat generation is at a maximum. Once there is a system shut down there is a need to know what's inside the reactor to make sure that it is safe to clean.

#### **4. Characterization of the actual products (and by-products; i.e., formation of undesirable products during the hydrolysis process) as a function of the extent of reaction:**

During the optimization of the process parameters in Phase II, samples will be taken to determine the products and by-products. This information will be used to optimize the hydrolysis process and to eliminate the formation of undesired products. Specifically:

- Products produced by the hydrolysis reactions will be fully characterize as a function of time to understand the reactions that are taking place during hydrolysis
- Picric Acid: The NRC has identified the formation of picric acid as a concern. Picric acid is formed from the degradation of tetryl at elevated temperature. Consequently, the potential of picric acid to be formed is always present if the hydrolysis reaction is incomplete and the environmental conditions are at elevated temperature.
- Stability of the hydrolysate: Continuation of the reactions after the hydrolysis of the energetics is completed is a concern of LANL, PANTEX and NSWC and needs to be addressed/understanding. The hydrolysate solution must be relatively stable and capable of being held for post-treatment processing. If the hydrolysate solution is not stable and reactions continue to occur, a release valve with a gas scrubber system should be investigated to insure there are no gas build up in the storage vessel.
- Final processing of the hydrolysate: Characterizing the hydrolysate for the next processing step is important. PANTEX noted significant solids in the hydrolysate. And, Pine Bluff Arsenal's SCWO for instance, can not inject slurries with particle sizes in excess of 100 microns.

In addition, as recommended by the NRC, whatever unit operations follows hydrolysis will be designed to accept emulsified nitro aromatic compounds

#### **5. Selection of chemical sensors and process control strategies to ensure those unit operations following hydrolysis can accept the product of hydrolysis:**

The chemical sensors and process control strategies will be developed during Phase I and evaluated during Phase II. After determination of all products and by products in Phase II, sensors and process control strategies will be refined and supplemented as necessary.

Phase III (optional) will be used to prove-out additional controls and control strategies for the supporting operations, if required by the PM.

#### **6. Development of a preventive maintenance (PM) program that minimizes the possibility of incidents during cleanups of accumulated precipitates:**

The kinetics study in Phase I will be used to assist in eliminating or minimizing the formation of undesirable products. Undesirable products could cause maintenance problems. The rate of build-up (or generation rate) of by-product salts that are potentially energetic will be assessed during testing, and a PM will be developed to prevent unnecessary operating conditions that may jeopardize worker safety. Trial runs will be performed during Phase II to determine the type of maintenance needed and the frequency. Material of construction will be investigated and chosen so that the material selected will resist alkaline, acid solutions, products and by-products of the hydrolysis.

- 7. Finding: The conditions under which aromatic nitro-compounds, such as TNT or picric acid, will emulsify in the aqueous phase and not be completely hydrolyzed are not well understood. Therefore, this type of material could be present in the output stream from an energetic hydrolysis:**

This will be studied during Phase I. In addition, the final product produced during Phase II will be analyzed for the presence of aromatic nitro compounds. If these compounds are detected, a plan will be formulated as to the best way to eliminate (or minimize) this by-product. Removal of these compounds from the final product (hydrolysate), if necessary, will be investigated in Phase III.

- 8. Finding: The products of hydrolysis of some energetic materials have not been characterized well enough to support simultaneous hydrolysis of different kinds of energetic material in the same batch reactor.**

As part of Phase I, the feasibility of simultaneous hydrolysis of different energetic materials in the same batch this will be investigated. However, safety issues may make this an unacceptable scenario. In addition and as recommended by the NRC, this program will investigate the formation of picrates from the hydrolysis of nitro aromatic compounds to assure they will not combined with M28 propellant.

## **APPENDIX C**

**Final Report, Alkaline Hydrolysis of Composition B, Tetrytol, M1, M8, and M28 Propellant, Los Alamos National Laboratory**

# Final Report

Alkaline Hydrolysis of Composition B, Tetrytol, M1, M8, and M28 Propellant

Robert Bishop and John Sanchez  
Los Alamos National Laboratory

*In order to further evaluate the use of alkaline hydrolysis for the first step in chemical weapons demilitarization, the hydrolysis of Composition B, Tetrytol, and M1, M8 and M28 propellant is currently being studied at a production scale (2000-gal batches) for the Program Manager for Assembled Chemical Weapons Activities (PMACWA) program. To support this study, 100-g sample of explosives and propellants were hydrolyzed in 12 and 20-wt% NaOH at 90-110°C. These studies were focused on measuring the hydrolysis reaction rate and product distribution, along with performing a general evaluation of the overall safety of the process.*

*The gaseous products were measured by online mass spectrometry. The liquid samples were analyzed in a batch fashion by ion chromatography (IC), total inorganic/organic carbon analysis (TOC), ammonia ion, high-pressure liquid chromatography (HPLC) and differential scanning calorimetry (DSC). The solid samples were studied using DSC and HPLC. The results of these and previous studies are presented in this paper along with some design suggestions for the production scale hydrolysis unit.*

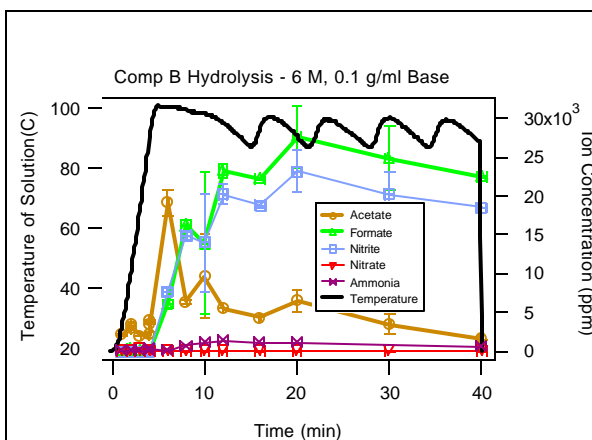
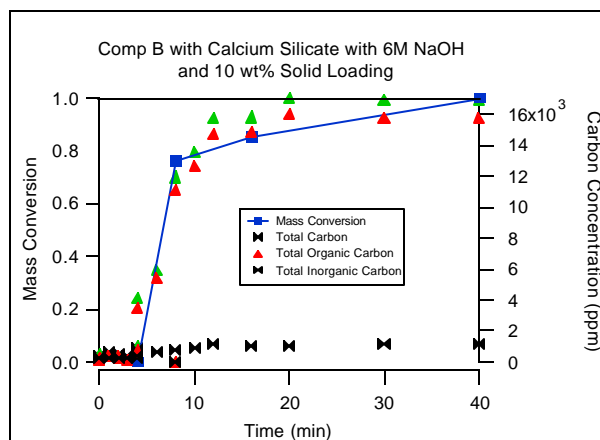
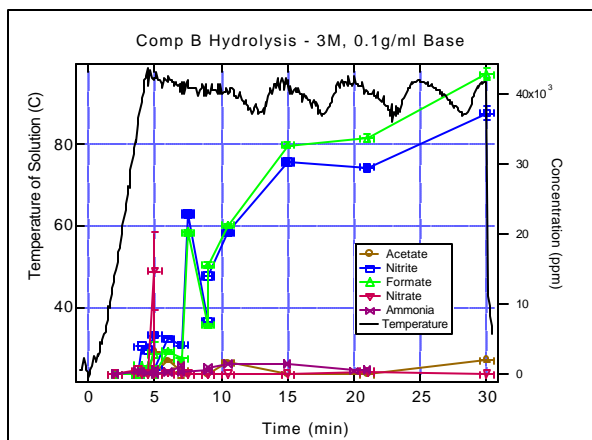
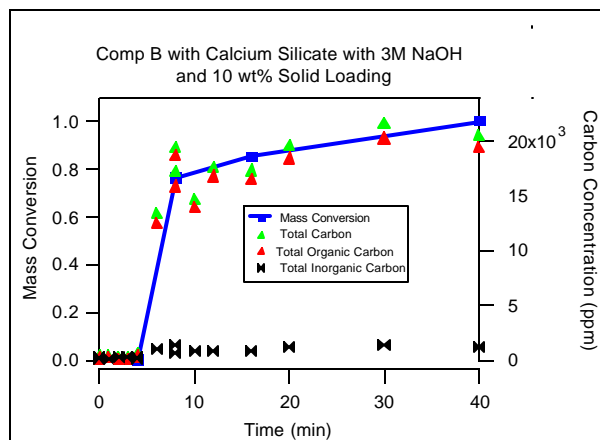
## **1.0 Introduction**

The United States and several other countries are actively developing programs in the field of chemical weapon demilitarization. The PMACWA program has been tasked with investigating several different technologies as alternatives to incineration. Many of these technologies will use base hydrolysis as the first step. In order to investigate base hydrolysis at an industrial scale, a 2000-gal reactor has been constructed at the Holston ammunition plant (Operated by Royal Ordnance North America). To support the operation of the large-scale reactor, bench-scale studies are being performed. This report presents those results.

## **2.0 Previous Alkaline Hydrolysis Studies**

### **2.1 PRELIMINARY EXPERIMENTS UNDER THE PMACWA PROGRAM**

Before the PMACWA experiments with the online mass spectrometer were performed, a few preliminary experiments were done. These experiments focused on the base hydrolysis of Composition B-4 (59.75% RDX, 39.75% TNT and 0.5% calcium silicate) in 50-ml batches. The results are shown in the figure and table on the next page. Only liquid and total mass analysis was performed. Each plot represents 4-5 experiments with different overall reaction times used in order to determine total mass conversion over time. The temperature trace is for the longest run time, but the temperature profiles were very consistent between experiments. The chief products were sodium formate and sodium nitrite with some sodium acetate, sodium nitrate and ammonia. The vast majority of the carbon in solution was organic (not sodium carbonate or sodium bicarbonate).



### Final Reaction Products

Starting NaOH (g/g Comp B)	NaNO <sub>2</sub> (g/g Comp B)	NaHCO <sub>2</sub> (g/g Comp B)	NaC <sub>2</sub> H <sub>3</sub> O <sub>2</sub> (g/g Comp B)	NH <sub>3(aq)</sub> (g/g Comp B)
1.2	0.31	0.34	0.037	0.030

## 2.2 Other Unpublished Or Recently Published Data From Los Alamos National Laboratory.

To aid in the analysis of the PMACWA results, results from previous experiments performed on TNT, and Tetrytol, were analyzed and reported below.

### 2.2.1 Composition B

Experiments in these tables used a 300mL liquid volume.<sup>1,2</sup>

#### Conversion Data

Experiment #	Mass Comp B	Temperature	Time at Temperature	Conversion*
1	20.49g (flakes)	90.3°C to 92.6 °C	300 sec	100% melted
2	0.54g (1-piece)	80.9°C	63 sec	100% melted
3	2g (1-piece)	81.7°C	140 sec	100% melted
4	2.35g (1-piece)	87.3°C	110 sec	100% melted
5	2.04g (1-piece)	88.5°C	60 sec	100% melted
6	3.12g (1-piece)	88.6 °C	100 sec	100% melted
7	3.05g (1-piece)	88.8°C	80 sec	100% melted
8	2.08g (1-piece)	89.2°C	65 sec	100% melted
9	1.99g (1-piece)	90°C	50 sec	100% melted
10	1.92g (1-piece)	91.3°C	35 sec	100% melted
11	13.67g (Several Pieces)	90.1°C	80 sec	100% melted

\* Some RDX Powder May Remain

#### 1.5 M NaOH Base Hydrolysis of Comp B

Experiment #	Mass Comp B	Temperature	Time at Temperature	Conversion
1	20.05g	90°C	6 min	17.9%
2	20.93g	90°C	12 min	64.6%
7	9.66g	90°C	30 min	97.7%
12	21.83g	83°C to 94.5°C	3 min	25%
13	21.97g	85°C to 94.5°C	5 min	57%
14	19.65g	85.5°C to 94°C	10 min	70%
15	2166g	85°C to 93.2°C	7 min	68%

#### 3 M NaOH Base Hydrolysis of Comp B

Experiment #	Mass Comp B	Temperature	Time at Temperature	Conversion
1	19.41g	90°C	3 min	12.4%
2	16.40g	90°C	6 min	22%
3	19.85g	90°C	12 min	56.4%
4	20.04g	90°C	18 min	74.8%
5	21.27g	90°C	6 min	15%
6	19.74g	90°C	18 min	68%

<sup>1</sup> Bishop, R. Unpublished Data – Los Alamos National Laboratory, DX-2 Division. 1/15/99.

<sup>2</sup> Bishop, R. L., Flesner, R. L., Larson, S. A., Bell, D. A., “Base Hydrolysis of TNT-Based Explosives.” *Journal of Energetic Materials*, **18**, 275-288 (2000).

### Ion Concentrations

Experiment	Acetate	Formate	Nitrite	Nitrate
1	2490 (4.2%C)	26050 (28.6%C)	27420 (28%N)	2730 (0.15%N)
2	1980 (3.3%C)	35900 (39.4%C)	38540 (39.3%N)	2160 (0.12%N)
4	460 (3.1%C)	1810 (7.3%C)	3540 (14%N)	200 (0.011%N)
1-neutralized	1230 (2.1%C)	33960 (34.4%C)	31270 (31.9%N)	1160 (0.063%N)
2-neutralized	1280 (2.1%C)	33270 (33.7%C)	30680 (31.3%N)	790 (0.043%N)

The average mass fractions are 0.011g/g for acetate, 0.10g/g for formate, 0.13g/g for nitrite, and 0.001g/g for nitrate.

### Gas Concentrations

In a previous study, the gas products from Composition B hydrolysis were measured. The measure molar ratios are 9.0scc/g for N<sub>2</sub>, and 46scc/g for N<sub>2</sub>O. However, the total gas volume was estimated by gas trapping over water and may not be accurate.

#### 2.2.2 Tetrytol

Experiments in these tables used a 300mL liquid volume.<sup>3,4</sup>

#### Conversion Data

#	Tetrytol Mass	Temperature	Time	Conversion	Mass Loading	[OH]
1	6.27 g (powder)	90°C	30 min	100%	10 wt%	1.5 M
2	58.63 g	90°C	5 min	17%	10 wt%	1.5 M
3	78.31 g	90°C	8 min	20%	10 wt%	1.5 M
4	68.71 g	90°C	16 min	43%	10 wt%	1.5 M
6	103.5 g	90°C	5 min	22%	10 wt%	1.5 M
7	94.66 g	90°C	6 min	16%	20 wt%	3.0 M
8	119.82 g	90°C	5 min	15.5%	10 wt%	1.5 M
9	76.15 g	90°C	5 min	15.7%	6 wt%	3.0 M
10	86.01 g	90°C	5 min	28%	10 wt%	3.0 M
11	95.49 g	90°C	5 min	12%	10 wt%	3.0 M
12	148.13 g	90°C	30 min	100%	15 wt%	3.0 M
13	102 g	90°C	23 min	90%	10 wt%	1.5 M

### Ion Concentrations

Experiment	Acetate	Formate	Nitrite	Nitrate
1	430 (0.47%C)	7260 (5.5%C)	48733(71.5%N)	250 (0.27%N)
2	60 (0.38%C)	463 (1.93%C)	2107 (17%N)	0 (0.0%N)
3	726 (4.0%C)	815 (2.9%C)	4125 (28.9%N)	993 (3.0%N)
1-neutralized	1573 (1.7%C)	7450 (5.64%C)	49657 (72.8%N)	187 (0.20%N)
2-neutralized	237 (1.5%C)	563 (2.34%C)	2847 (23.0%N)	47 (0.28%N)
3-neutralized	767 (4.22%C)	2727 (9.7%C)	810 (5.67%N)	743 (2.24%N)
Completion	270 (0.49%C)	3290 (3.9%)	23480 (54.7%)	87 (0.15%N)

<sup>3</sup> Bishop, R. Unpublished Data – Los Alamos National Laboratory, DX-2 Division. 1/15/99.

<sup>4</sup> Bishop, R. L., Flesner, R. L., Larson, S. A., Bell, D. A., “Base Hydrolysis of TNT-Based Explosives.” *Journal of Energetic Materials*, **18**, 275-288 (2000).



The average molar ratios are 0.002g/g for acetate, 0.017g/g for formate, 0.16g/g for nitrite, and 0.0006g/g for nitrate.

### 2.2.3 Tetryl Hydrolysis

Tetryl has not been hydrolyzed separately at LANL. However, tetrytol and TNT have been treated separately. Therefore, some inferences can be drawn about the hydrolysis of tetryl alone. The only structural difference between tetryl and TNT is that a nitramine replaces the methyl on the ring. At first glance, this difference should result in a larger percentage of nitrous oxide, nitrogen gas and sodium nitrite to be produced during the reaction.

The table below compares the results for tetrytol and TNT.<sup>5</sup>

Species	TNT Hydrolysis	Tetrytol Hydrolysis	% Difference
<u>Carbon</u>			
Formate (g/g)	0.11	0.065	41
Formate (%C)	8	6	25
<u>Nitrogen</u>			
Nitrite (g/g)	0.21	0.23	9.5
Nitrite (%N)	34	32	5.9
Ammonia (scc/g)	30	102	240

The comparison shows that the hydrolysis of tetryl is very similar to that of TNT. The additional nitrite is consistent with the larger percent of nitrogen in the tetryl molecule (24%) compared to TNT (19%). There is a measurable difference in formate production between the two molecules. However, the low percentage of carbon in the molecule converted to measurable products (<10%) makes it difficult to quantify any possible difference between the mechanism of tetryl and TNT hydrolysis. Furthermore, some of the difference is also due to the lower fraction of carbon (29%) in tetryl compared to TNT (37%).

The only clear difference in hydrolysis products for tetry and TNT, is the quantity of ammonia gas produced. This could be due to the either the additional nitrogen in the tetryl molecule or the use of a mass spectroscopy to measure the ammonia concentration for the tetryl versus measuring ammonia by capturing the hydrolysis off-gas in an acid solution for the TNT. Further research on tetry and TNT needs to be performed to better evaluate this apparent difference.

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<sup>5</sup> Bishop, R. L., Flesner, R. L., Larson, S. A., Bell, D. A., "Base Hydrolysis of TNT-Based Explosives." *Journal of Energetic Materials*, **18**, 275-288 (2000).

### 3.0 New Results under the PMAWA Studies

#### 3.1 *EXPERIMENTAL SPECIFICS*

##### 3.1.1 Materials

The explosives and propellants were provided through Picatinny Arsenal and the PMACWA program. The compounds studied were Comp B-4 (59.75% RDX, 39.75% TNT, and 0.5% Calcium Silicate ), Tetrytol (70% tetryl and 30% TNT ), M1 Propellant (85% nitrocellulose, 10% DNT, 5% dibutylphthalate, 1% diphenylamine), M8 propellant (52.15% nitrocellulose, 43% nitroglycerin, 1.25% potassium nitrate, 3% diethylphthalate, 0.60% ethyl centralite), and M28 propellant (60% nitrocellulose, 23.8% nitroglycerin, 9.9% triacetin, 2.3% dimethylphthalate, and 2.2% lead stearate ). The Comp B-4 was provided as large flakes, the Tetrytol was in small to medium chunks, the M1 propellant was in 30-35 g sheets, and the M1 and M8 propellants were small grains.

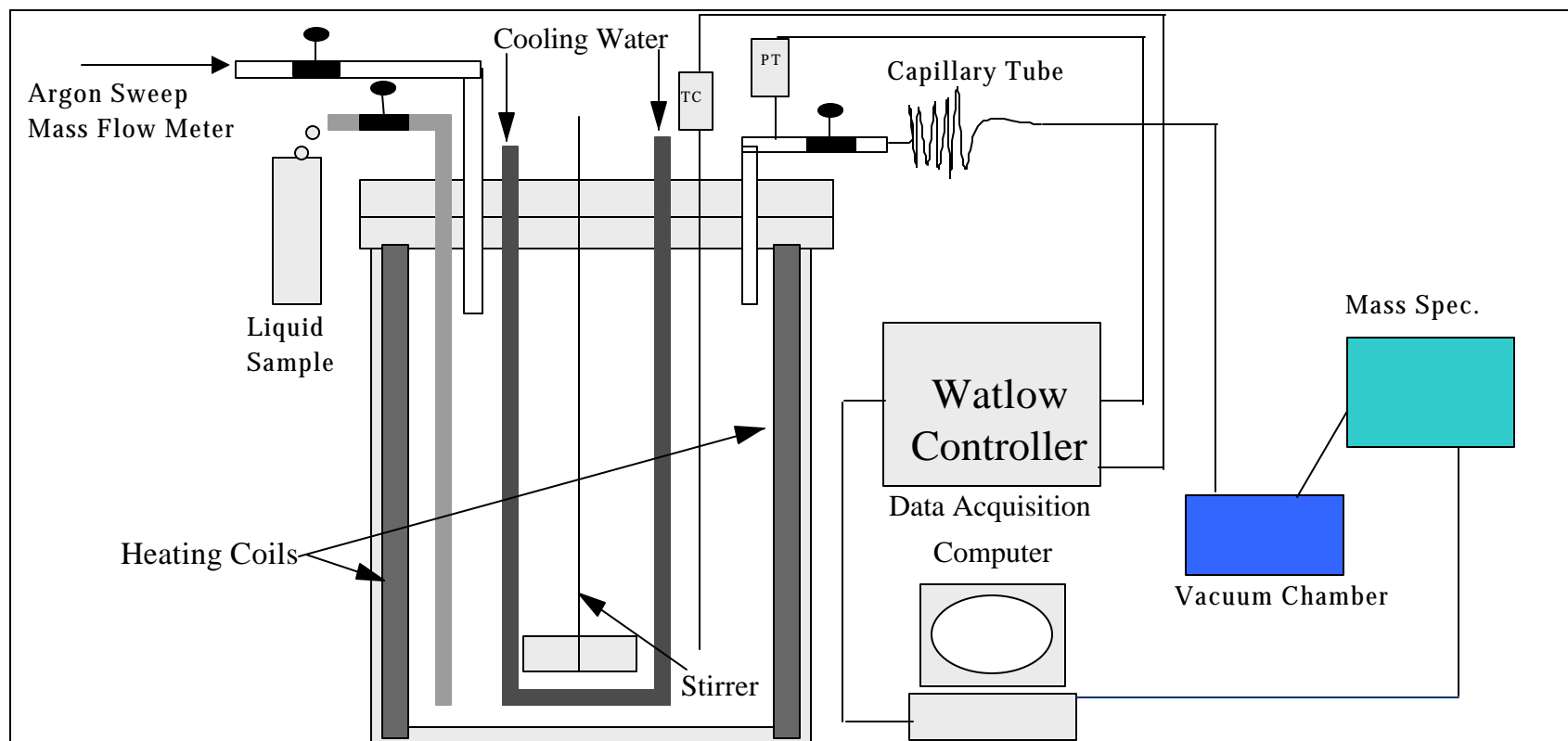
The sodium hydroxide was made using dry-pellets of NaOH (over 98.5% pure) and house de-ionized water.

##### 3.1.2 Two Liter Parr Reactor Assembly

The experiments were performed in a 2L stainless steel reactor (Parr Instrument Company). A schematic of the reactor assembly is shown on the following page. Electric coil heaters in contact with the reactor walls heated the reactor and an internal cooling coil using chilled water provided cooling. A PID controller (Watlow Series 945) maintained a set reaction temperature. The data acquisition program (Labview, National Instruments) recorded temperature, pressure, and stirrer shaft rotational speed every five seconds. It was noted in later experiments that the stirrer shaft was periodically slipping out of alignment; this might have caused the actual stirrer speed to be slower than indicated.

The presence of a sampling port allowed liquid samples to be taken during the experimental run. The liquid samples were diluted 10x in cold water to stop the reaction and kept in a refrigerator until processing. The gas products were analyzed using an online mass spectrometer. The product gases were swept into the mass spectrometer inlet using argon gas.

## Base Hydrolysis Reactor (2 Liter Parr)



### 3.1.3 Analytical Methods

#### 3.1.3.1 Ion Chromatography

The anions nitrite ( $\text{NO}_2^-$ ), formate ( $\text{HCOO}^-$ ), nitrate ( $\text{NO}_3^-$ ) and acetate ( $\text{CH}_3\text{COO}^-$ ) were analyzed using a Dionex AI 450 Ion Chromatograph Analyzer equipped with an AS 11 ION Pac Column, guard column, and anion self-regenerating suppressor. A conductivity detector measured the ion concentrations. Three eluents: de-ionized water, 5mM NaOH, and 100 mM NaOH were mixed to create an elution concentration gradient with time. The elution flow rate was 2.5mL/min. Calibration test using a mixture of 1-ppm standards were used for calibration prior to analysis.

The analysis done by Dale Counce at EES-6, (LANL) also detected oxalate ( $\text{C}_2\text{O}_4^{2-}$ ) and a peak that is possibly glycolate (. The oxalate measurements were included in the results for Composition B-4, Tetrytol, and M28/Composition B-4.

#### 3.1.3.2 On-line Mass Spectrometer

A Balzers Quadstar<sup>TM</sup> mass spectrometer was used for the on-line gas analysis. The Ion current (A) was recorded as a function of time for the following mass to charge ratios: 4, 14, 15, 16, 17, 18, 27, 28, 30, 32, 36, 40, and 44. The individual gas partial pressures were calculated using the following mass charge ratio assignments:  $\text{N}_2$  (28),  $\text{O}_2$  (32),  $\text{N}_2\text{O}$  (44), Ar (40),  $\text{NH}_3$  (15), and NO (30). When calculating the partial pressure of  $\text{N}_2$ , 10.8% of the magnitude of peak 44 was subtracted from the magnitude of peak 28 to compensate for the  $\text{N}_2$  fragment generated from  $\text{N}_2\text{O}$ . Furthermore, when calculating the partial pressure of NO, 31.1% of the magnitude of peak 44 was subtracted from the magnitude of peak 30 to compensate for the NO fragment generated from  $\text{N}_2\text{O}$ . These values were taken from the Balzer's fragmentation library included in the Quadstar<sup>TM</sup> software package.

The mass spectrometer was calibrated by following calibration gases past the capillary tube opening until equilibrium was reached. The calibration gases were: 1) 78.08%  $\text{N}_2$ , 20.95%  $\text{O}_2$ , 0.93% Ar and 0.03%  $\text{CO}_2$ ; 2) 1.007%  $\text{N}_2\text{O}$  and 98.993% Ar; 3) 0.993% Ar, 8.115%  $\text{CO}_2$ , 7.925% CO, 2.006%  $\text{H}_2$ , 0.5064%  $\text{N}_2\text{O}$ , balance  $\text{N}_2$ ; 4) 100%  $\text{NH}_3$ ; 5) 5.033%  $\text{CO}_2$ , balance Ar; 6) 0.4851% NO, balance  $\text{N}_2$ . Full calibrations were performed periodically with an air calibration check performed before each day's experiments.

#### 3.1.3.3 Total Organic Carbon/Total Inorganic Carbon

A Rosemount Dohrmann Total Organic Carbon Analyzer DC-190 was used to determine the total organic and total inorganic carbon (TOC/TIC) in the hydrolysate solution. A 50mM sample, containing a 1:10 dilution of hydrolysate solution to de-ionized water was used. The digestion buffer was 20wt% phosphoric acid.

#### 3.1.3.4 Ammonia Probes

The ammonia concentration was measured with an Orion ammonia electrode and the Orion 290A pH meter. The electrode was calibrated using 0.005 and 0.0005M NH<sub>4</sub>OH standards.

#### 3.1.3.5 Thermocouples

The thermocouples were Omega type J thermocouples. Type J is iron and copper-nickel. This type is well suited for the temperature ranges studied (20°C – 160°C). Dual thermocouples were used on the Parr reactor, one for control and one as a high limit temperature switch.

#### 3.1.3.6 Pressure Transducers

The pressure was measured using Omega pressure transducers. The pressure transducer was read using an Omega DP41-E-A model pressure monitor. For these experiments the pressure was near atmospheric (sweep gas passed through an open check valve during the run) and is not presented in this report. However, the pressure data was recorded in a data file during the experiment.

#### 3.1.3.7 Gas Flow Meters

The flow rate of the argon sweep gas was controlled at 200 standard cubic centimeters per minute using a Mass Flow Meter/Controller (Scott Specialty Gases). The calibration was performed at the factory prior to shipping.

### 3.2 *RESULTS*

#### 3.2.1 Comp B (59.75% RDX, 39.75% TNT, and 0.5% Calcium Silicate)

In the first experiment 99.82g of Comp B-4 was added to 1kg of 12wt% NaOH. The stirrer was set at 710 RPM. There was no Comp B-4 remaining at the end of the experiment. The reactor temperature reached 93 °C at 2500 sec and the reactor was held at 93 °C until 6000 sec.

In the second experiment 81.48g of Comp B-4 was added to 1kg of 20wt% NaOH. The stirrer was set at 720 RPM. There was no Comp B-4 remaining at the end of the experiment. The temperature profile is presented with the other results.

#### 3.2.2 Tetrytol (70% Tetryl and 30% TNT)

In the first experiment 100.29g of tetrytol was added to 1kg of 12wt% NaOH. The stirrer was set at 710 RPM. There was no tetrytol remaining at the end of the experiment. The temperature profile is presented with the other results.

In the second experiment 91.16g of tetrytol was added to 1kg of 20wt% NaOH. The stirrer was set at 708 RPM. There was no tetrytol remaining at the end of the experiment. The temperature profile is presented with the other results.

### 3.2.3 M1 Propellant (85% nitrocellulose, 10% DNT, 5% dibutylphyhalate, 1% diphenylamine)

In the first experiment 100-g of M1 propellant was added to 1kg of 12wt% NaOH. The stirring motor was set at 704 RPM. There were 17g of residue remaining after the experimental run due to incomplete reaction (some propellant grains were remaining).

In the second experiment 100- g of M1 propellant was added to 1kg of 20wt% NaOH. The stirring motor was set at 704 RPM. There were 7g of residue remaining after the experimental run was completed (no propellant grains remaining but some oily, tar-like residue on the filter paper).

### 3.2.4 M8 Propellant (52.15% nitrocellulose, 43% nitroglycerin, 1.25% potassium nitrate, 3% diethylphthalate, 0.60% ethyl centralite)

The first experiment was performed on 111.3g (3 bundles) of M8 propellant in 1kg of 20wt% NaOH. The stirring motor was set at 712 RPM. There were 6.3g of residue remaining after the experimental run was completed (some propellant grains did remain but the majority of the mass was from residue on the filter paper). The strings that tied the bundles together caused entanglement problems around the stirrer shaft, this may have led to some of the propellant not reacting. The resultant solution and solid residue has a strong odor similar to white pepper.

The second experiment was performed with 109.8g (3 bundles) of M8 propellant in 1kg of 20wt% NaOH. The stirring motor was set at 712 RPM. There were 4.5g of residue remaining after the experimental run was completed (no propellant grains remaining but residue on the filter paper). The strings that tied the bundles together did not degrade and may cause entanglement problems. The resultant solution and solid residue has a strong odor similar to white pepper.

### 3.2.5 M28 Propellant (60% nitrocellulose, 23.8% nitroglycerin, 9.9% triacetin, 2.3% dimethylphthalate, and 2.2% lead stearate)

The first experiment was performed with 99.56g of M28 propellant in 1kg of 12wt% NaOH. The stirring motor was set at 711 RPM. There were 6.52g of residue remaining after the experimental run was completed.

The experiment was performed with 100.45g of M28 propellant in 1kg of 20wt% NaOH. The stirring motor was set at 710 RPM. There were 5.14 g of residue remaining after the experimental run was completed.

### 3.2.6 M28 Propellant with Tetrytol (40% M28. 60% Tetrytol)

The first experiment was performed with 100g of M28 propellant and Tetrytol in 1kg of 12wt% NaOH. The stirring motor was set at 710 RPM. There were 3.73g of residue remaining after the experimental run was completed.

The second experiment was performed with 100g of M28 propellant and Tetrytol in 1 kg of 20wt% NaOH. The stirring motor was set at 709 RPM. There was 4 g of residue remaining after the experimental run was completed.

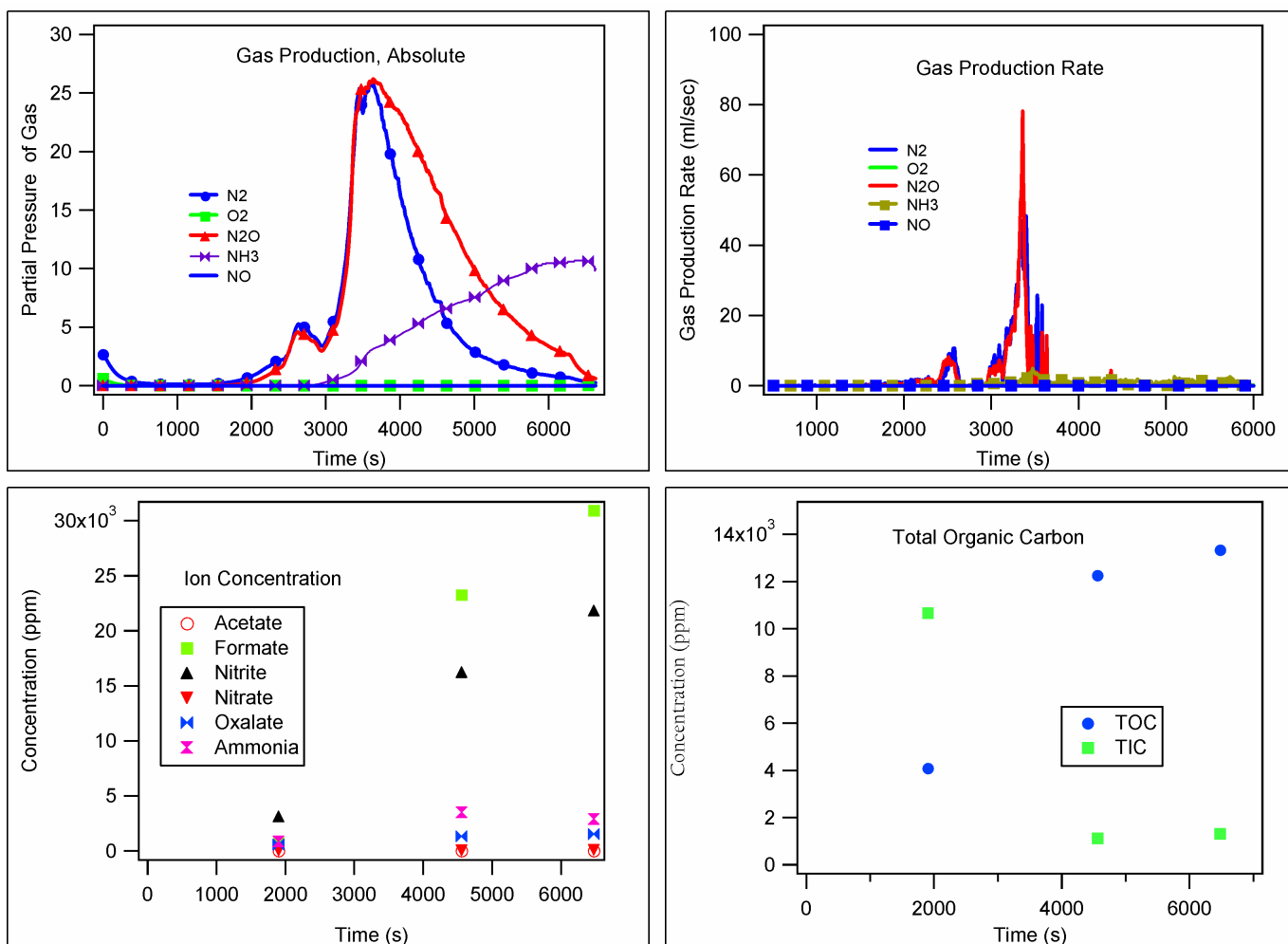
### 3.2.7 M28 Propellant with Composition B-4

The first experiment was performed with 86g of M28 propellant and 14.5 g of Composition B-4 in 1kg of 12wt% NaOH. The stirring motor was set at 712 RPM. There was 21.72 g of residue and propellant remaining after the run

The second experiment was performed with 86.17g of M28 propellant and 14.25g of Composition B-4 in 1kg of 20wt% NaOH. The stirring motor was set at 712 RPM. There were 2.86g of residue remaining after the run

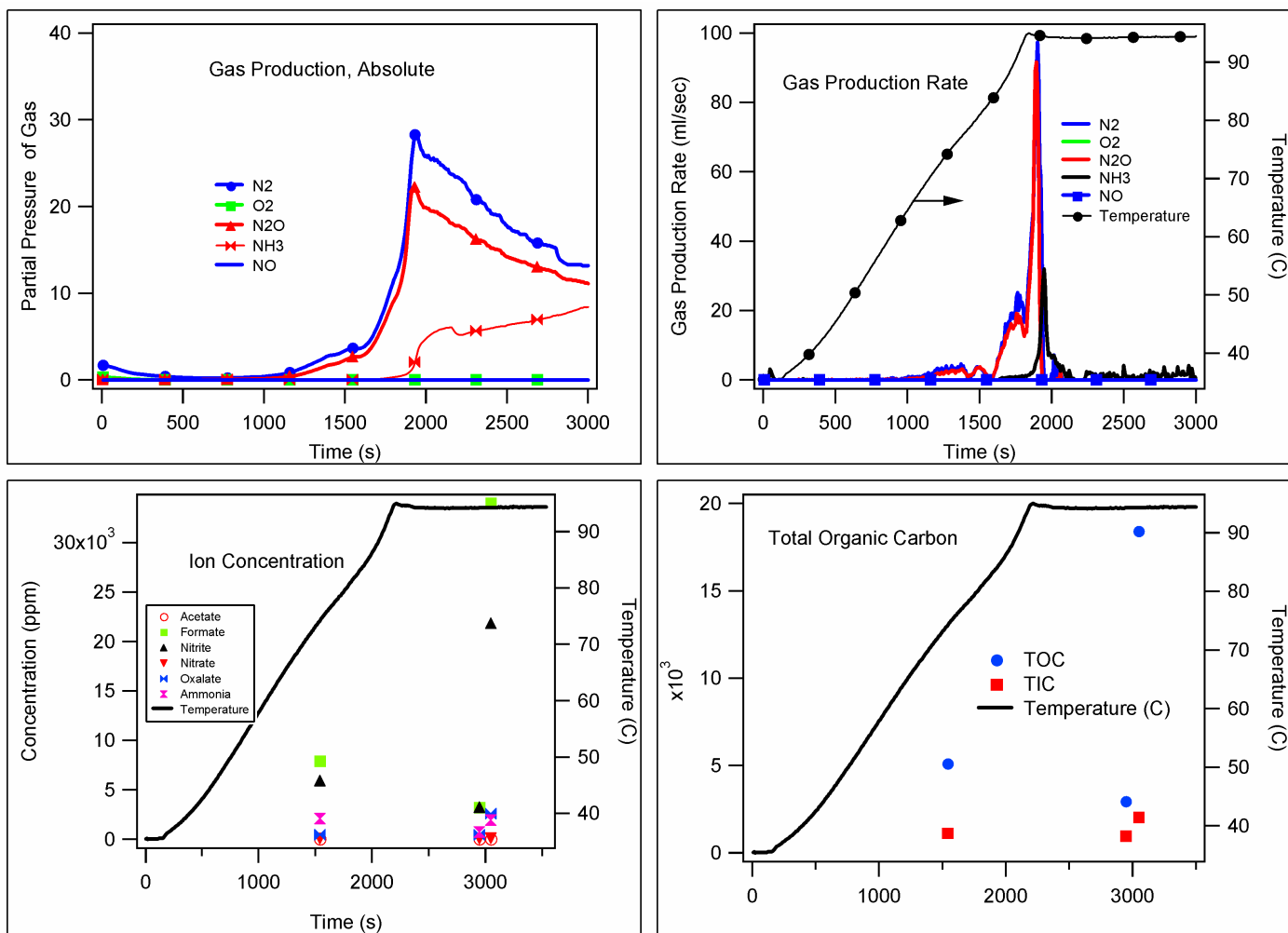
### 3.3 Gas Concentration/Aqueous Products Plots

#### 3.3.1 A) Composition B-4 in 12wt% NaOH

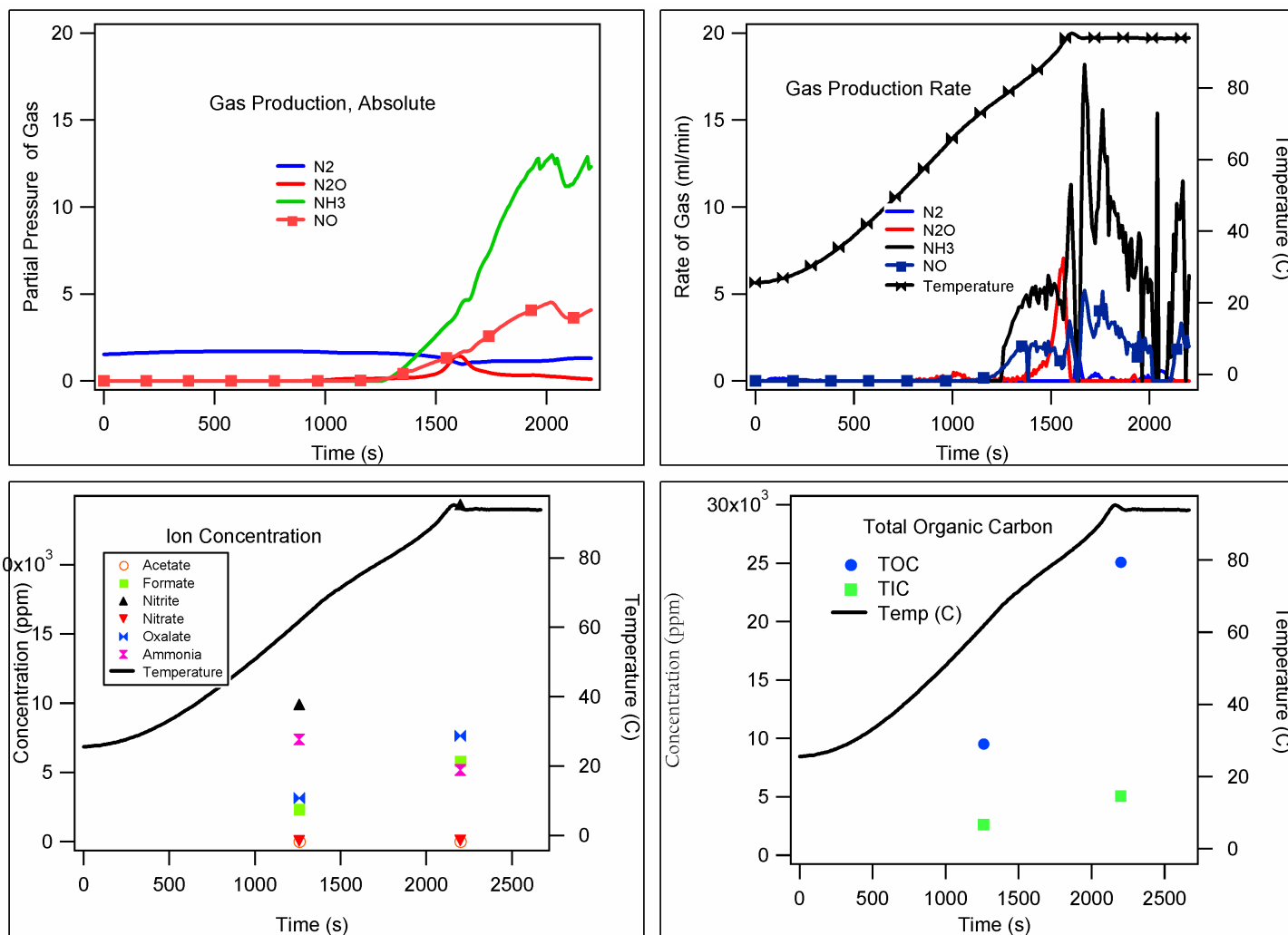




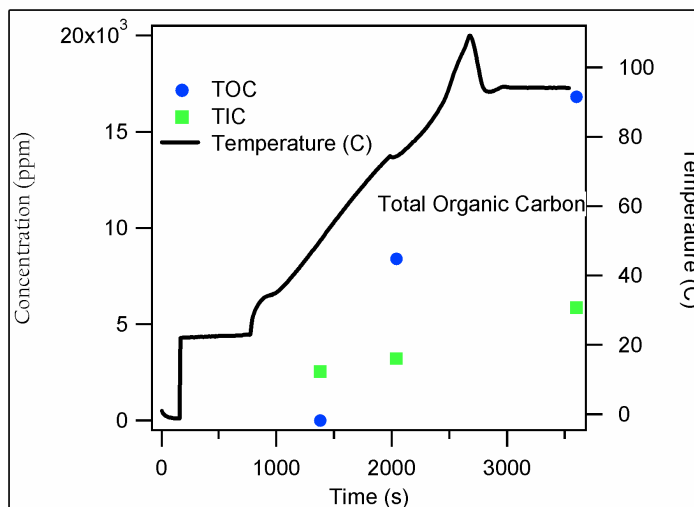
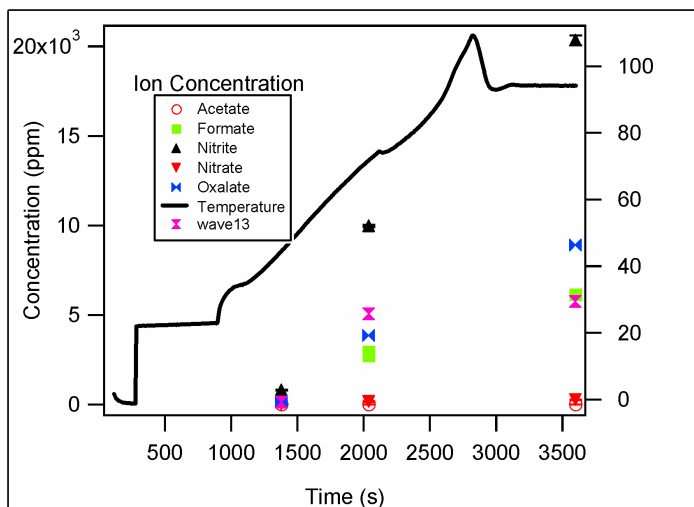
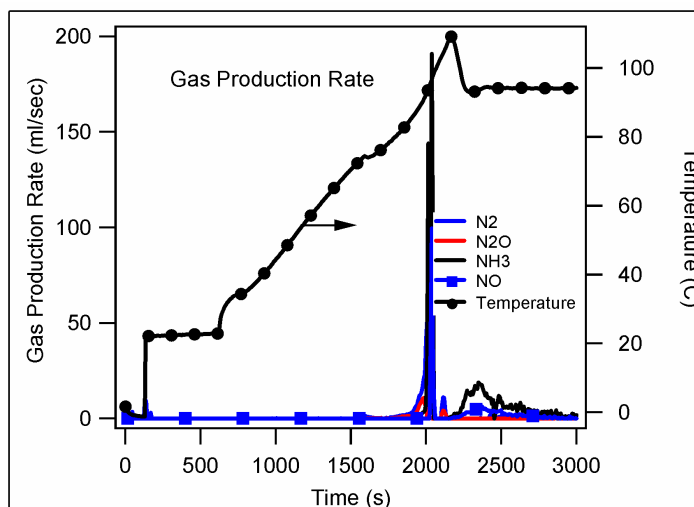
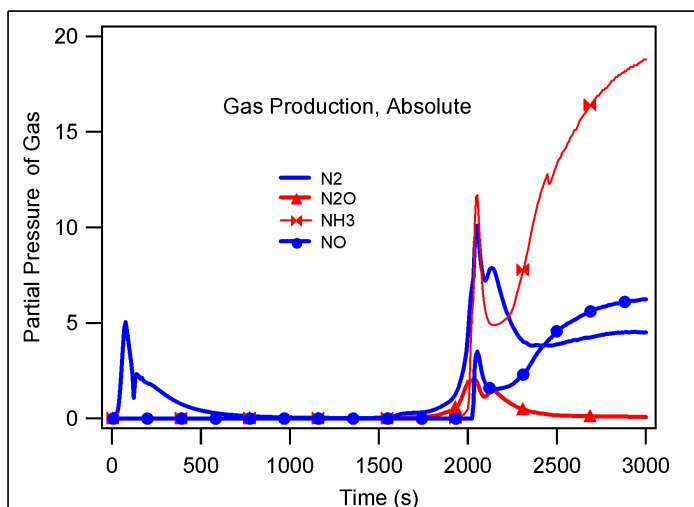
### 3.3.1 B) Composition B-4 in 20 wt% NaOH



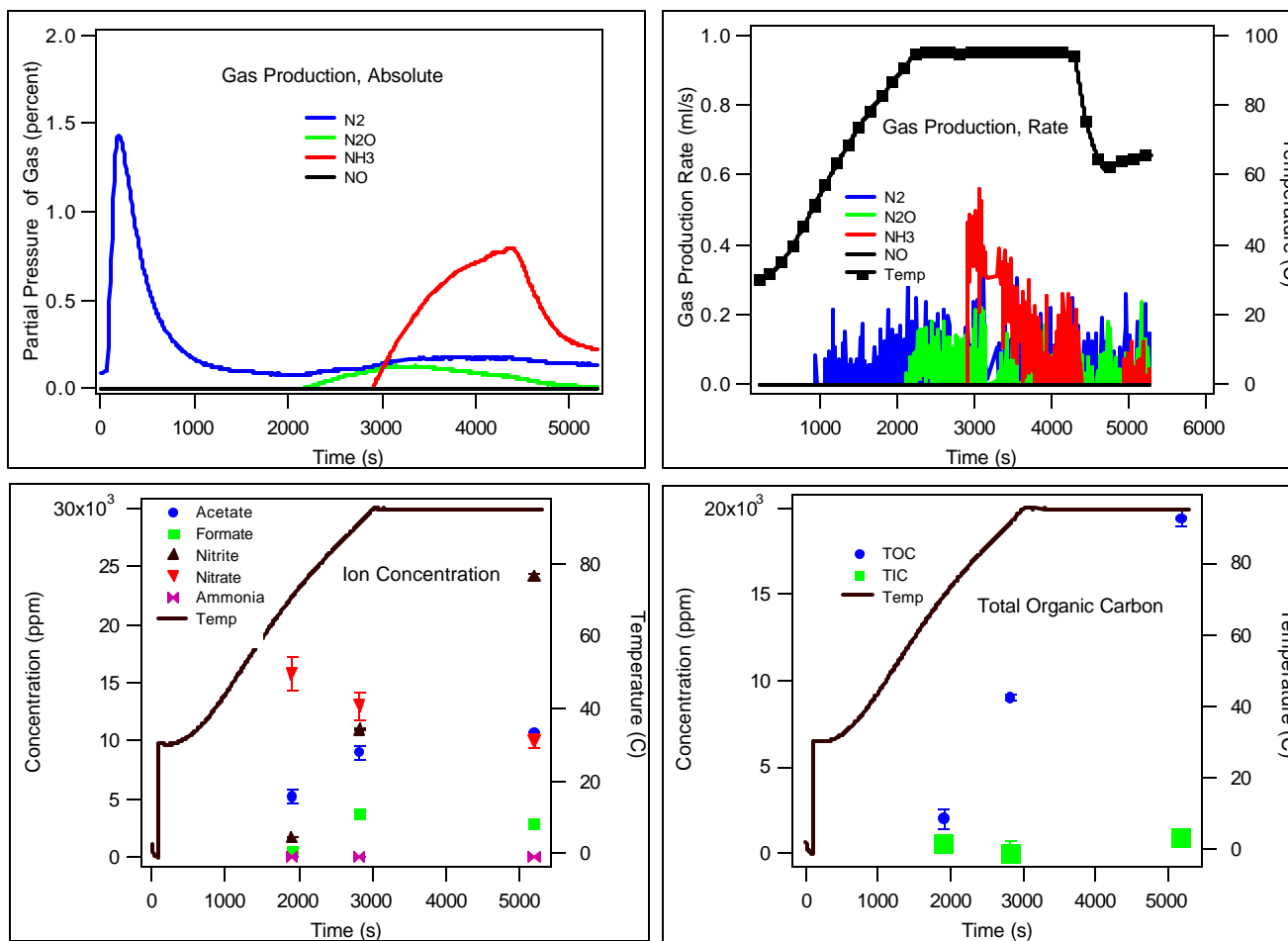
### 3.3.2 A) Tetrytol in 12 wt% NaOH



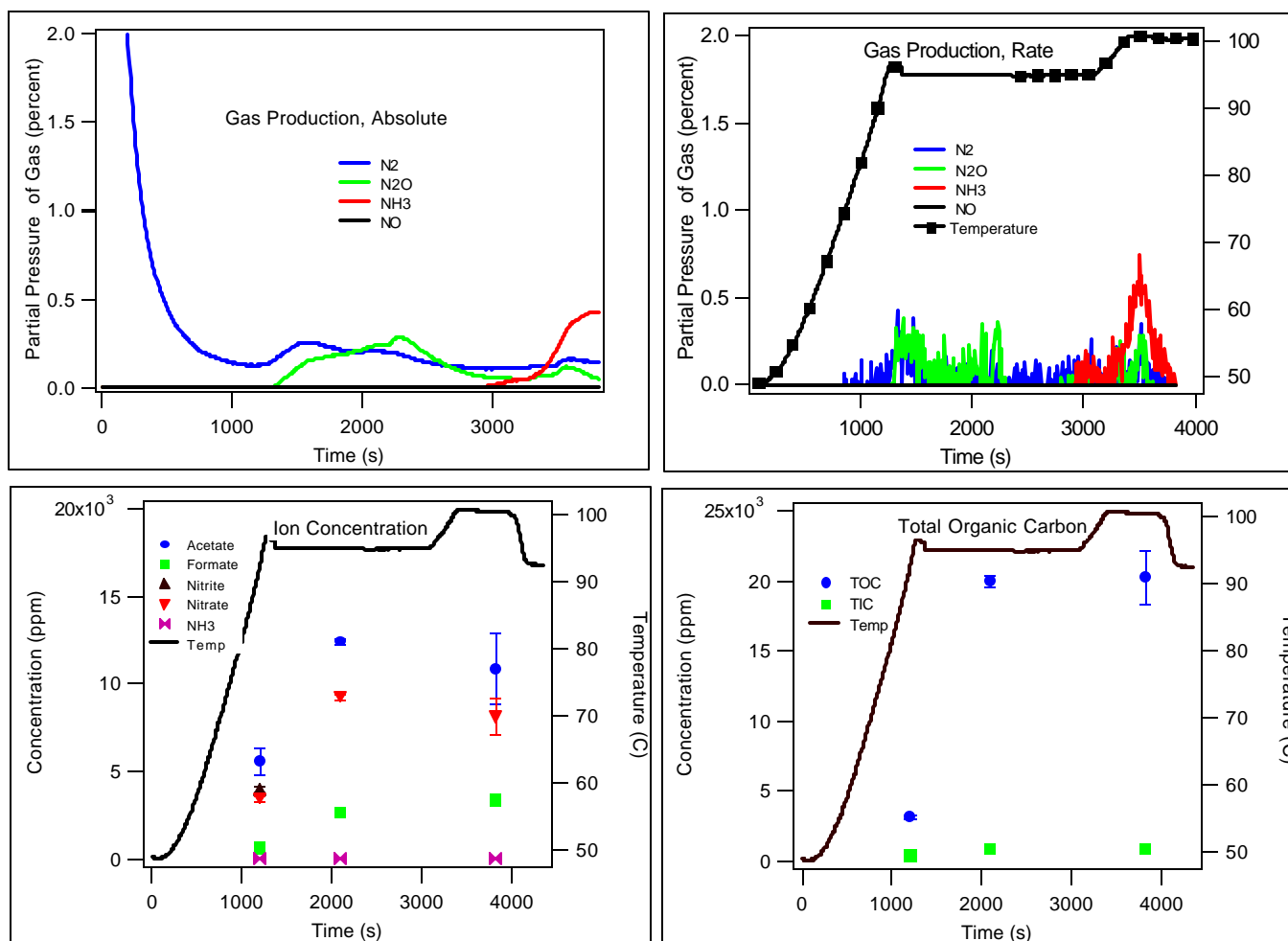
### 3.3.2 B) Tetrytol in 20 wt% NaOH



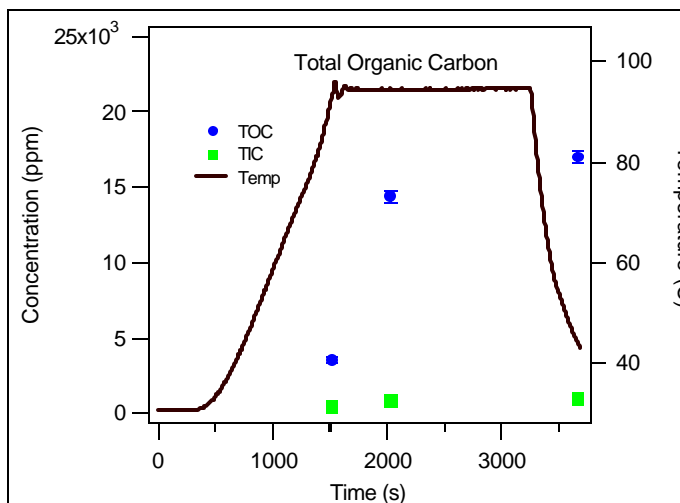
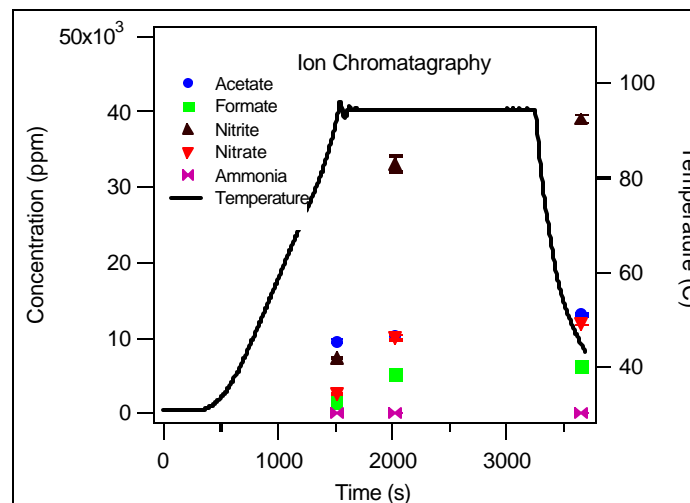
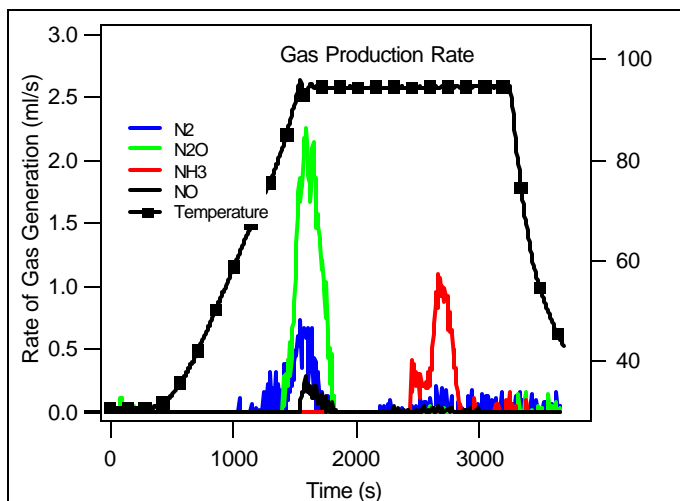
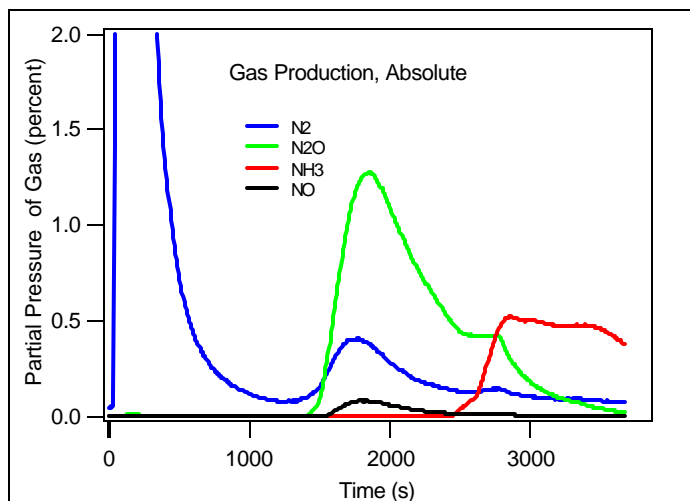
### 3.3.3 A) M1 Propellant in 12 wt% NaOH



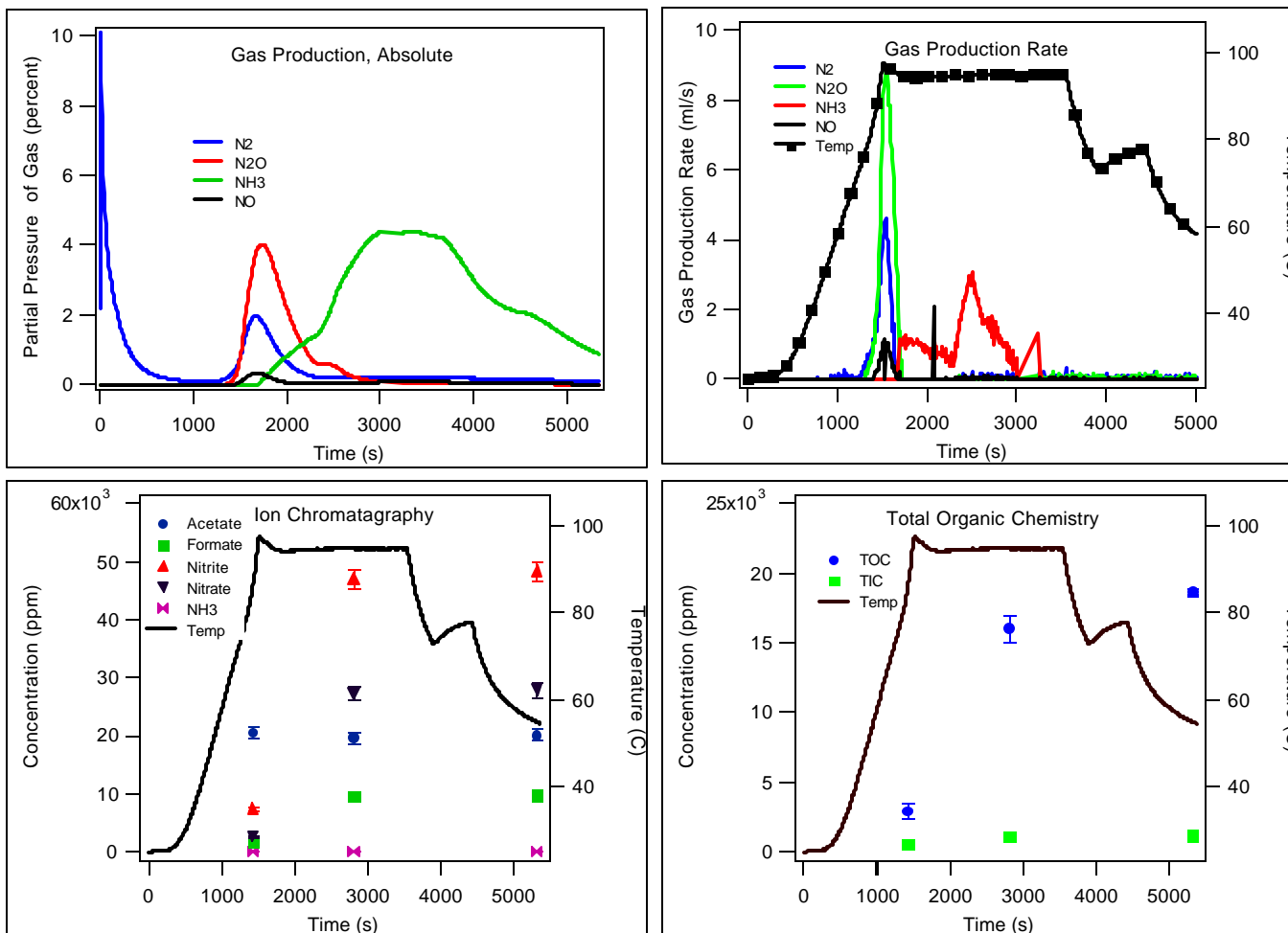
### 3.3.3 B) M1 Propellant in 20 wt% NaOH



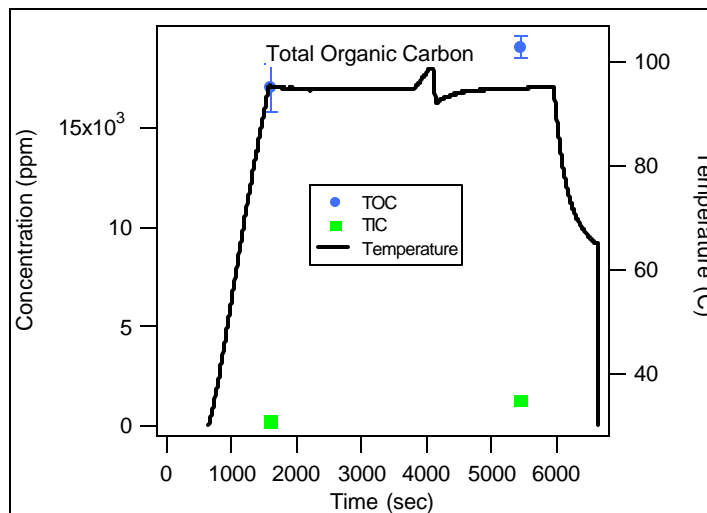
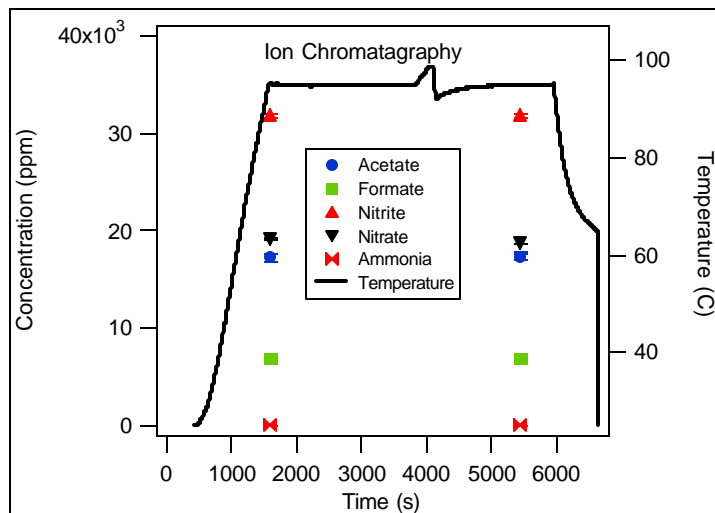
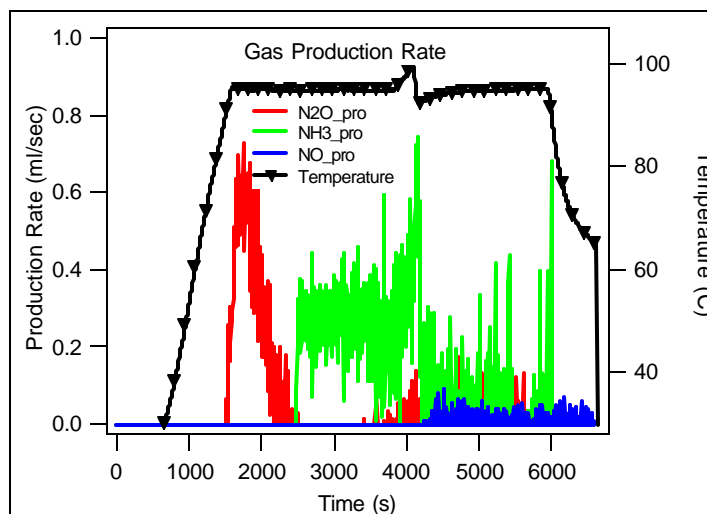
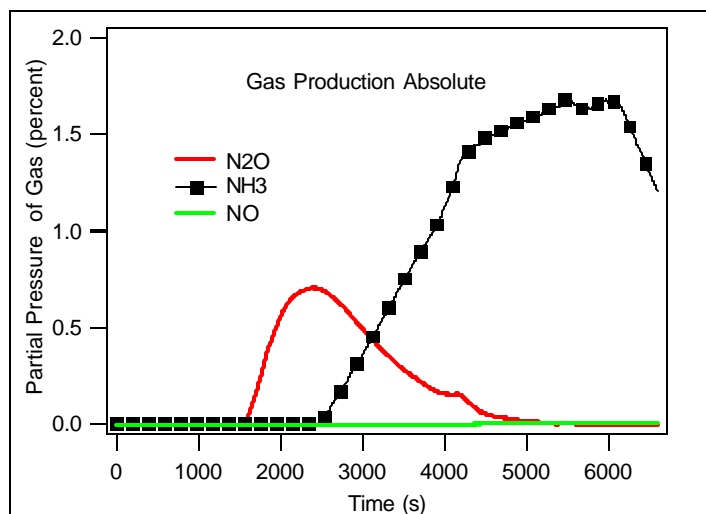
### 3.3.4 A) M8 Propellant in 12 wt% NaOH



### 3.3.4 B) M8 Propellant in 20 wt% NaOH

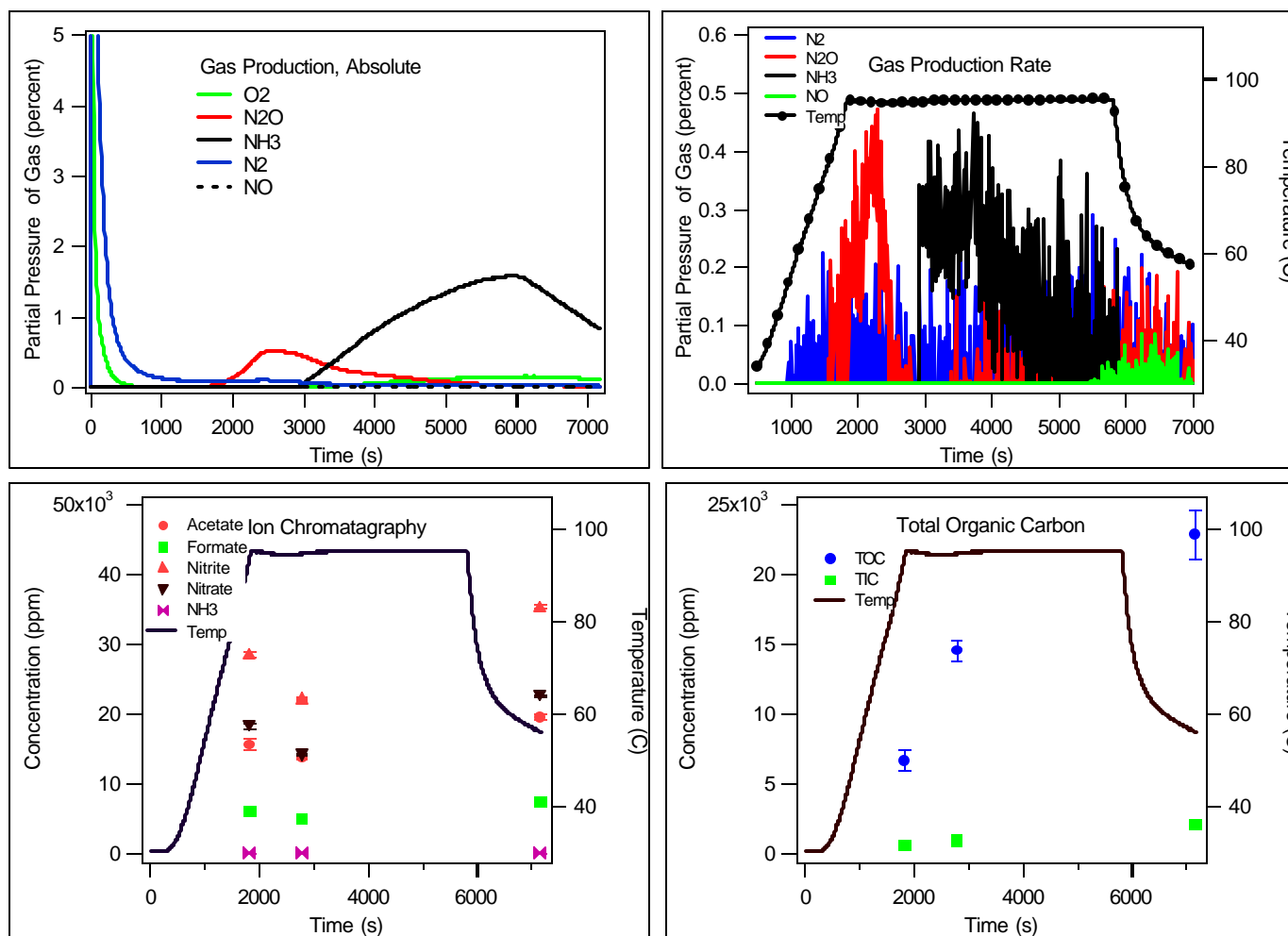


### 3.3.5 A) M28 Propellant in 12 wt% NaOH

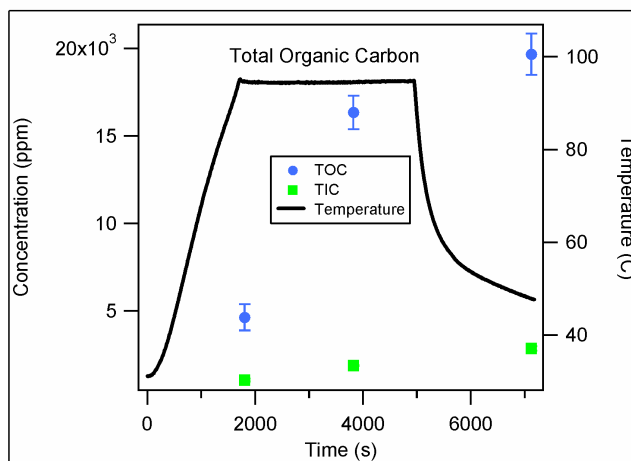
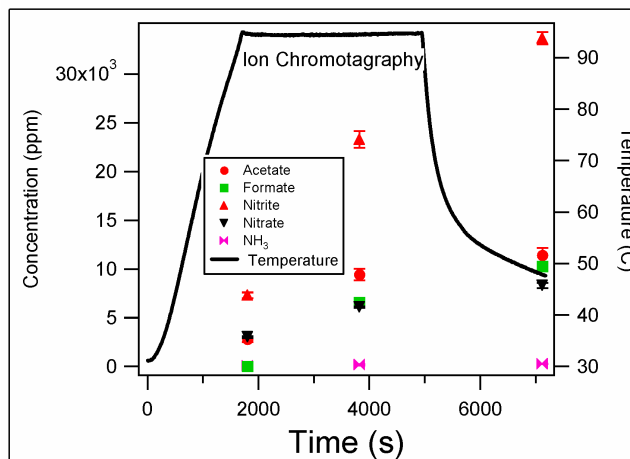
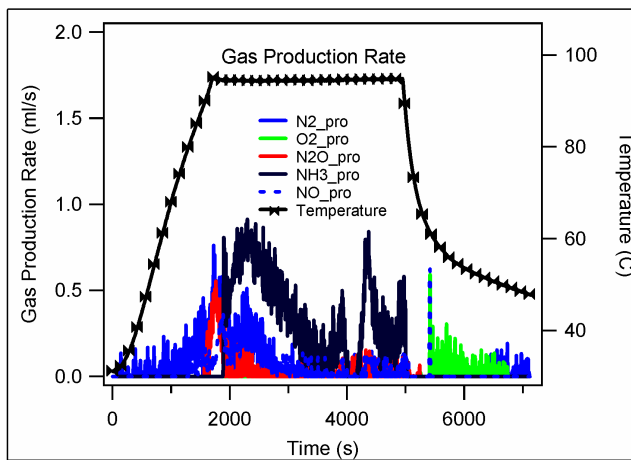
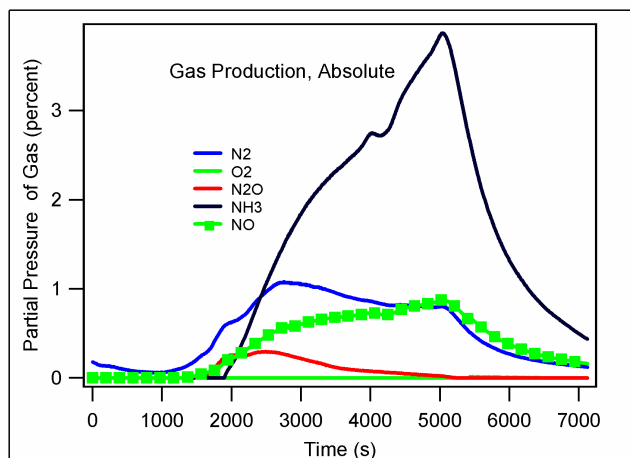




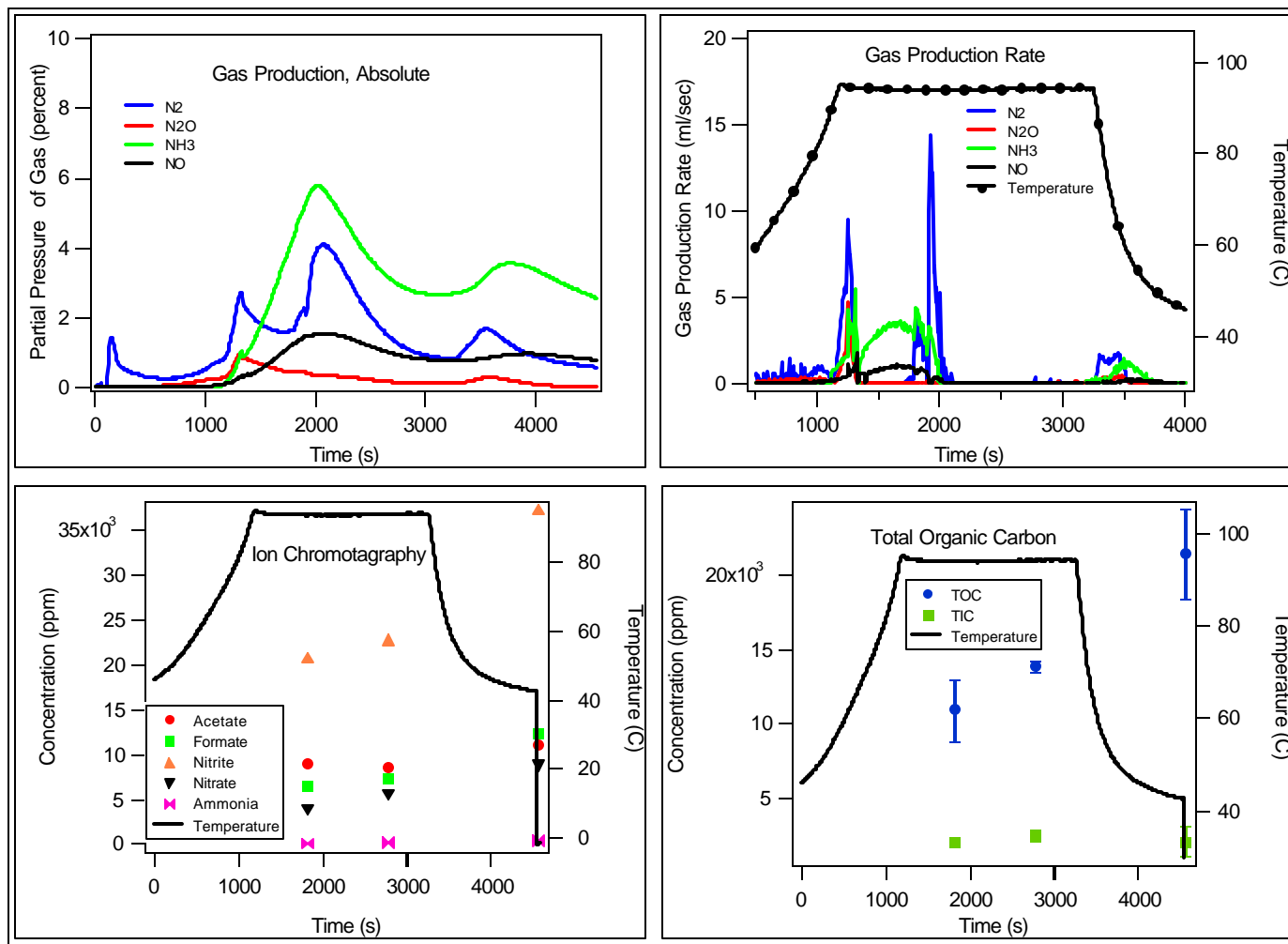
### 3.3.5 B) M28 propellant in 20wt% NaOH



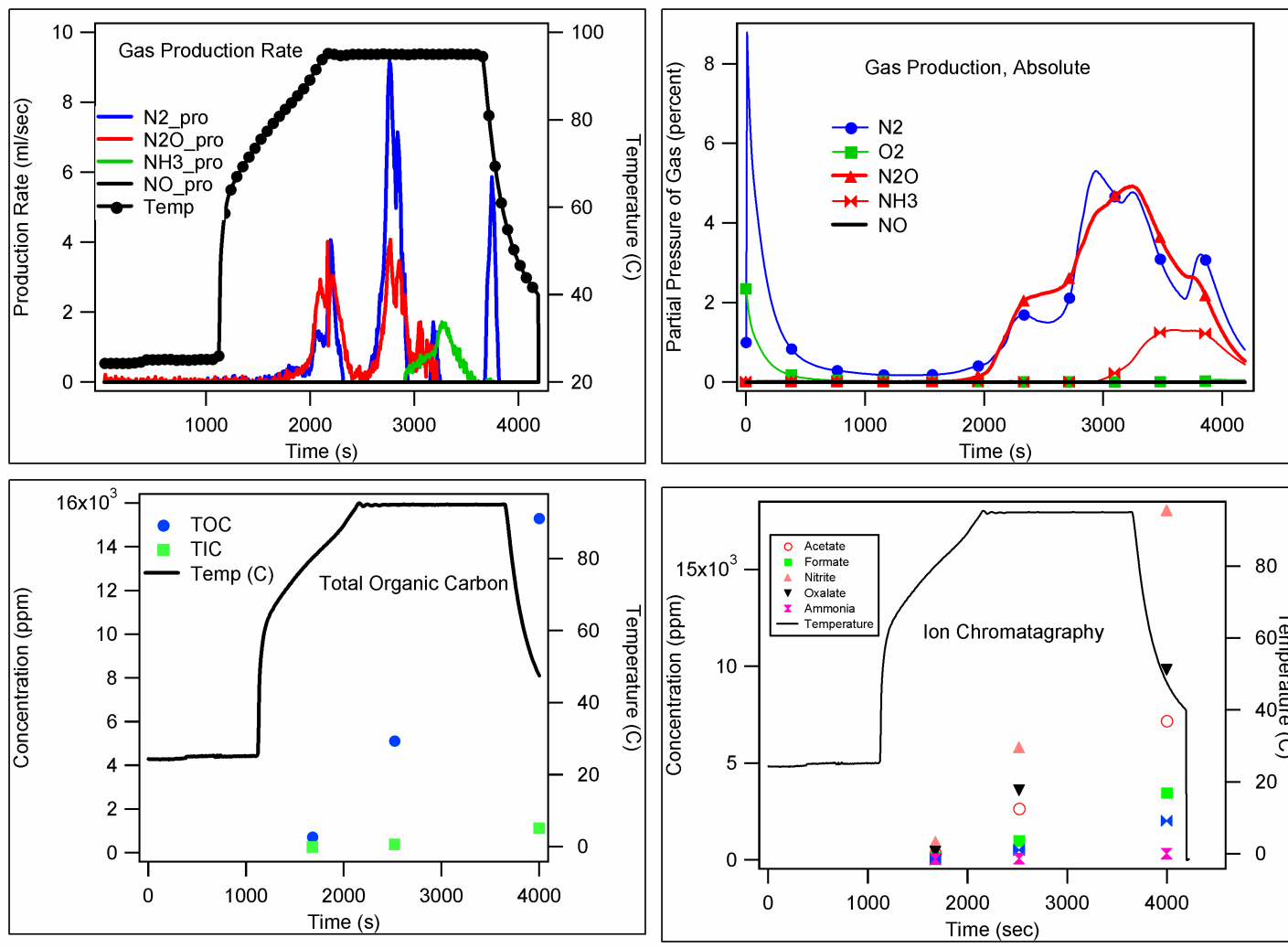
### 3.3.6 A) M28 Propellant with Tetrytol in 12 wt% NaOH



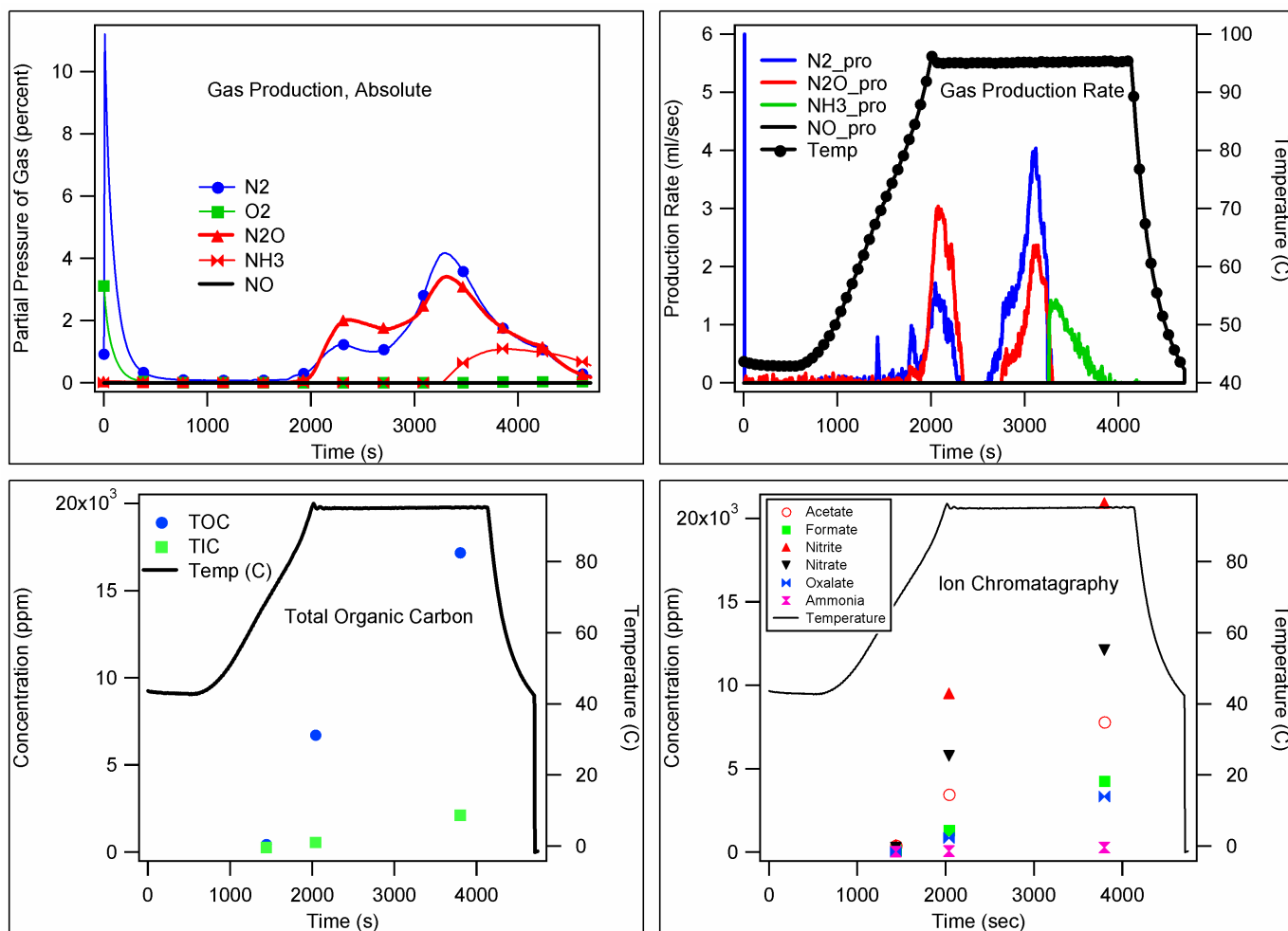
### 3.3.6 B) M28 Propellant with Tetrytol in 20 wt% NaOH



### 3.3.7 A) M28 Propellant with Composition B-4 in 12 wt% NaOH

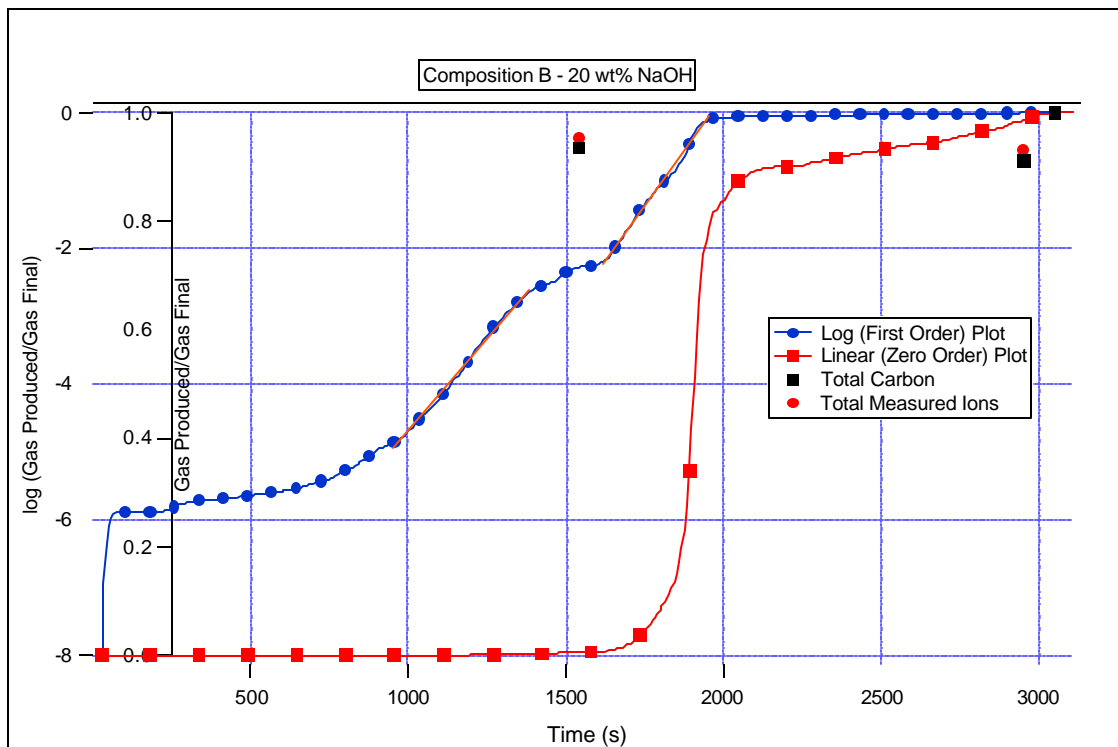
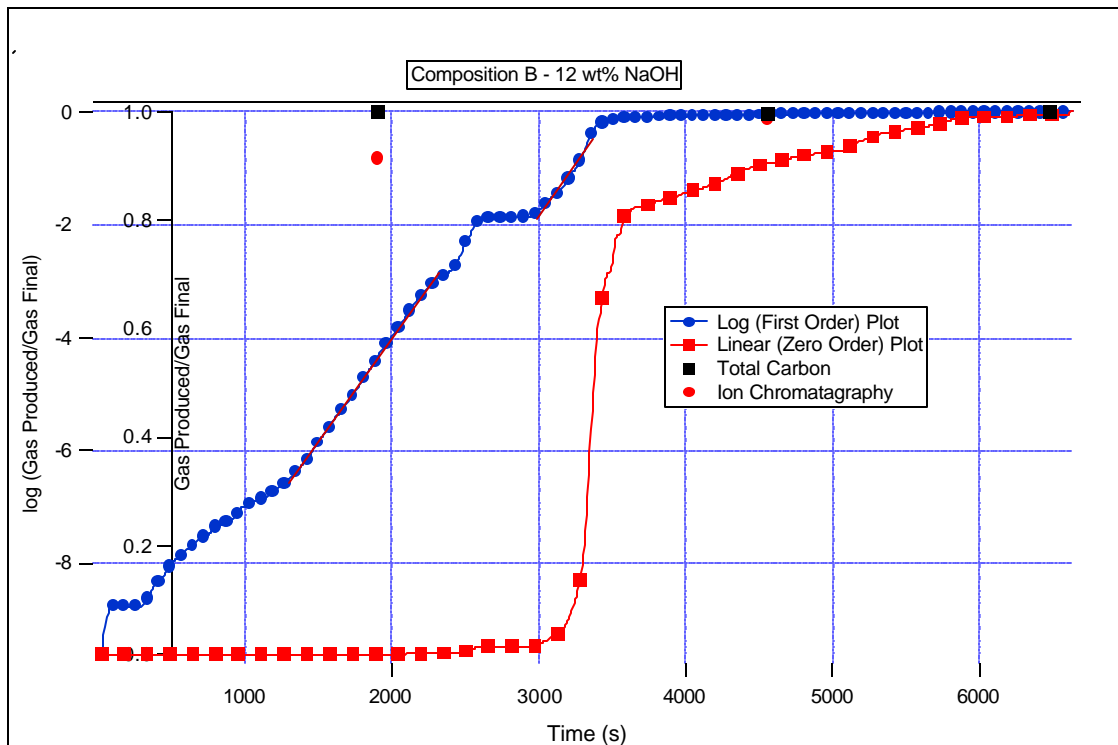


### 3.3.7 B) M28 Propellant with Composition B-4 in 20 wt% NaOH

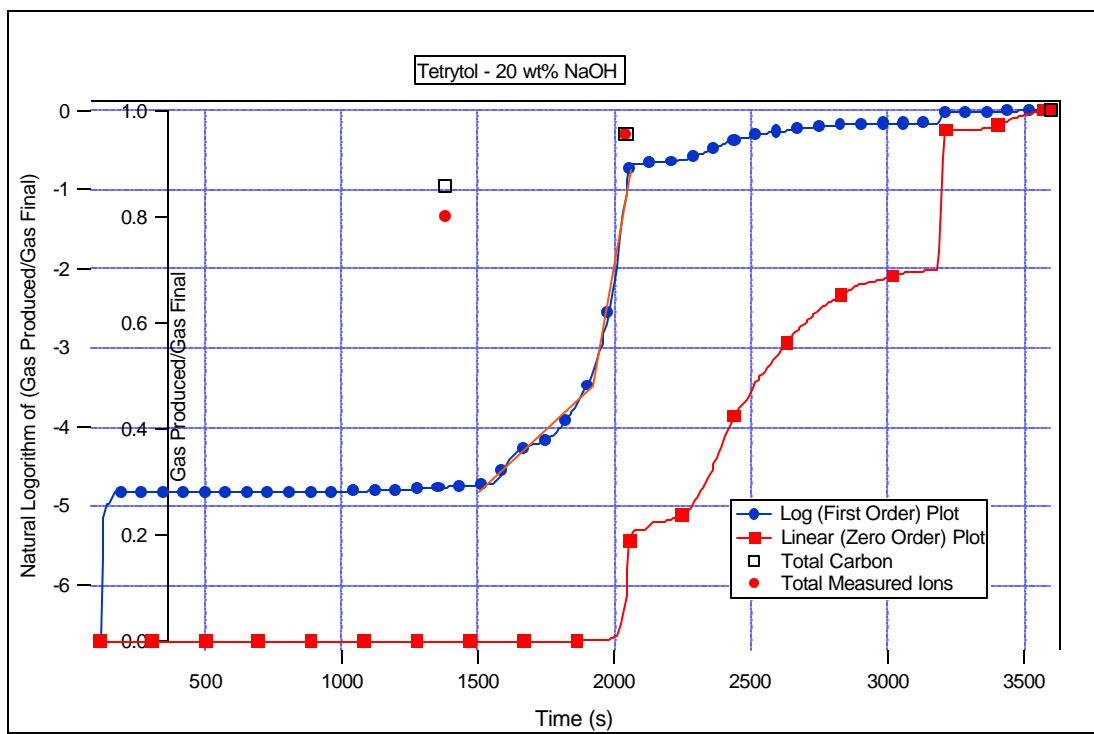
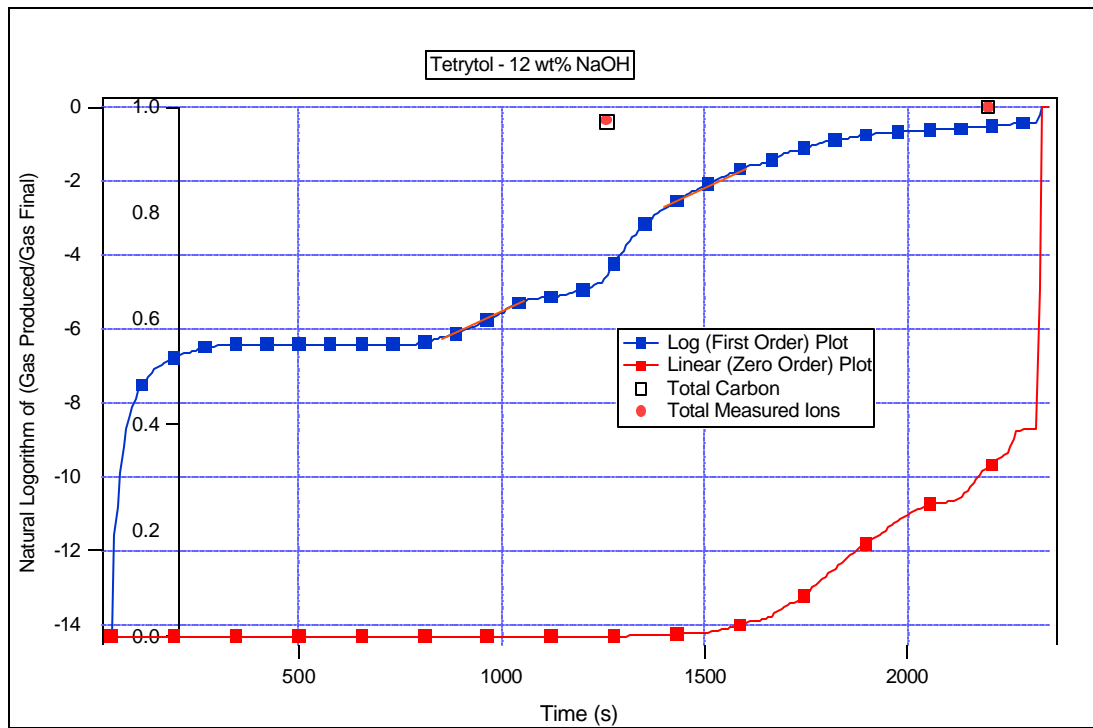


### 3.3 GAS PRODUCTION RATE PLOTS (IC AND TOC DATA ARE ON A LOG BASIS)

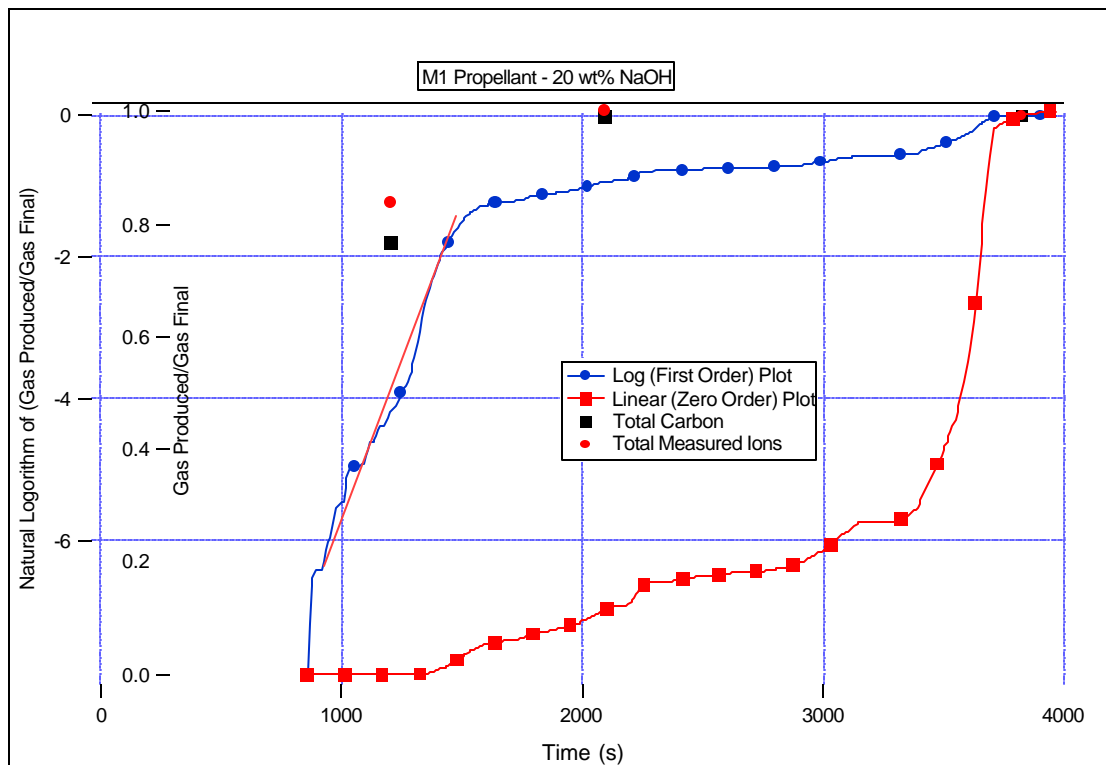
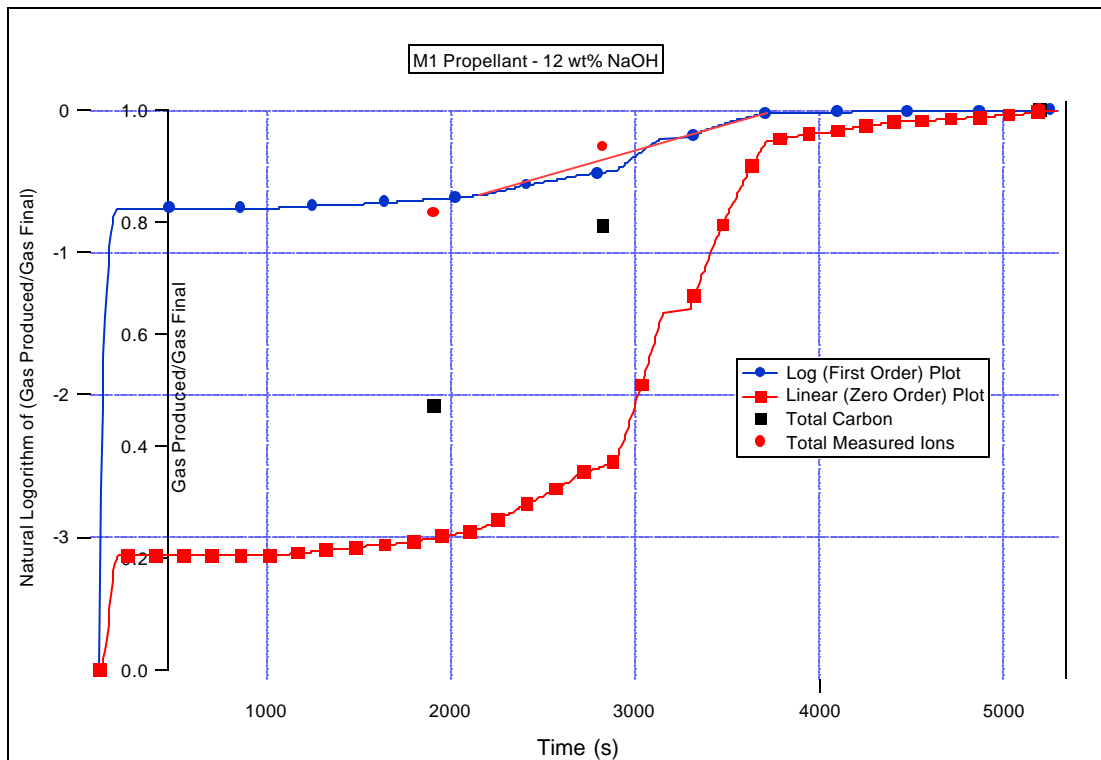
#### 3.3.1 Composition B



#### 3.3.2 Tetrytol

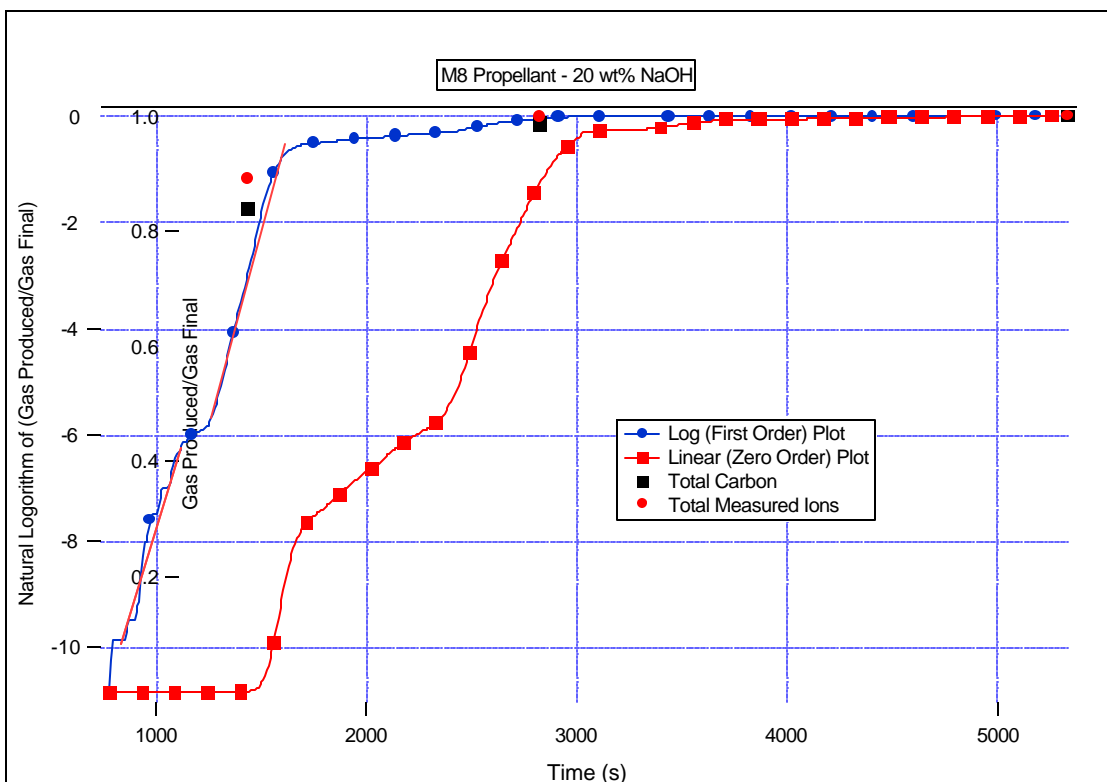
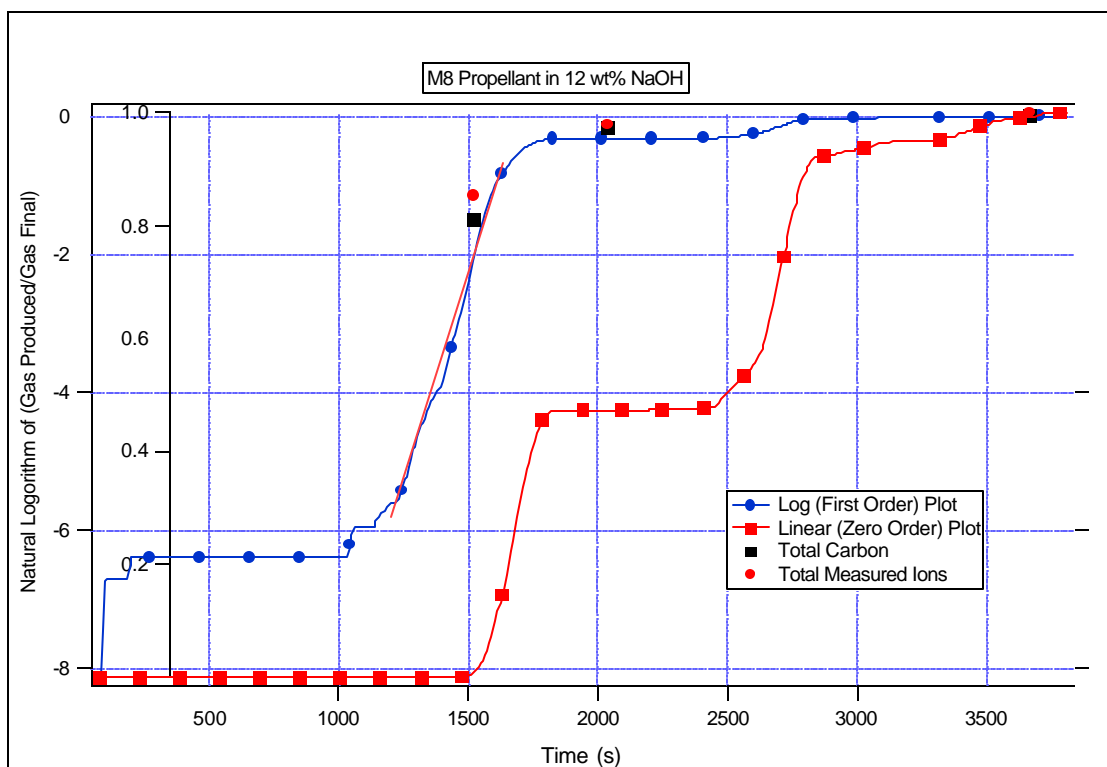


### 3.3.3 M1 Propellant

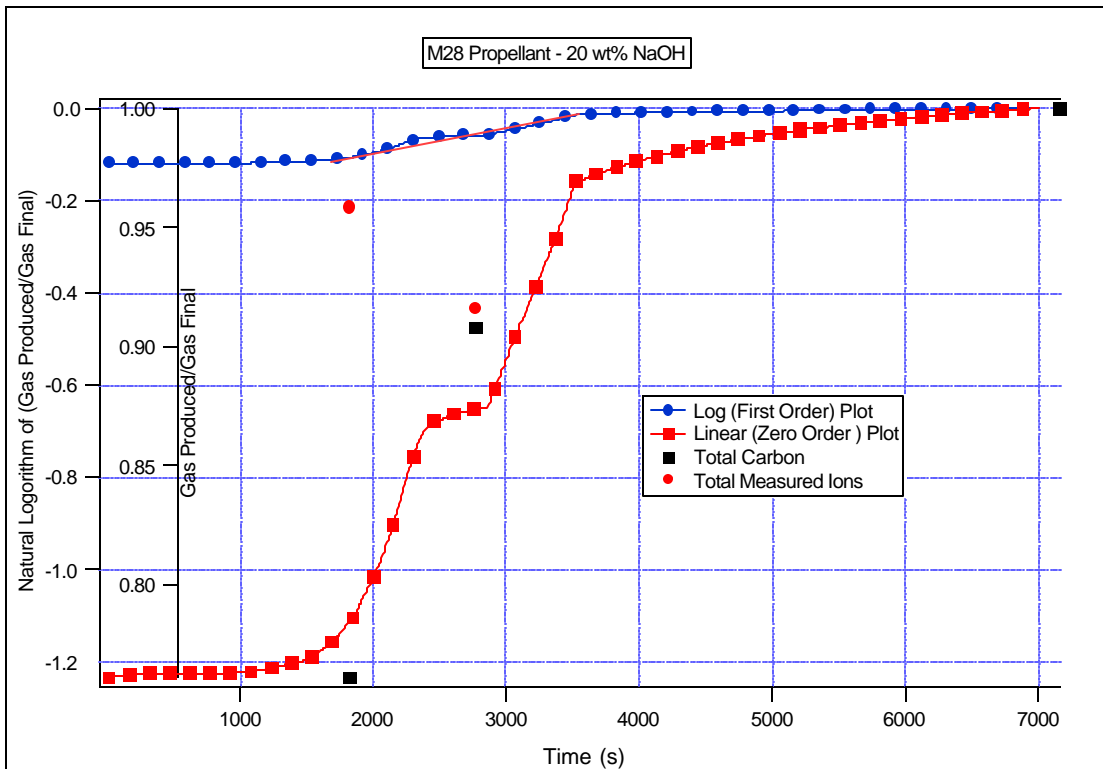
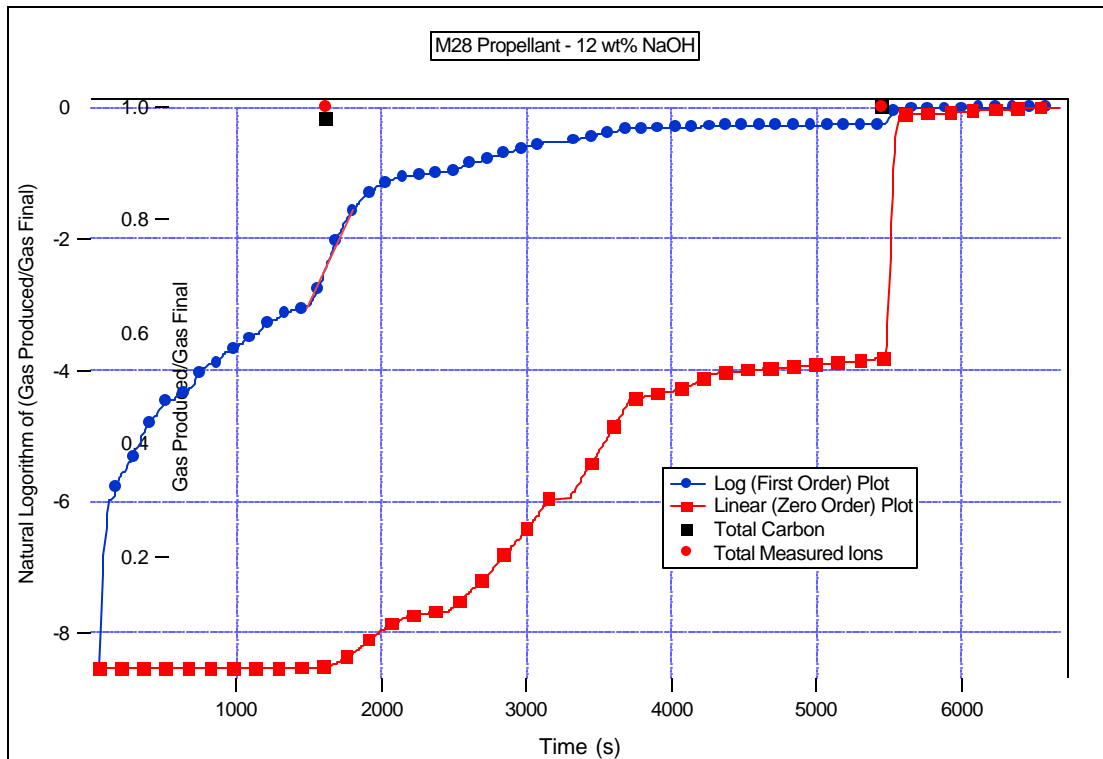


### 3.3.4 M8 Propellant

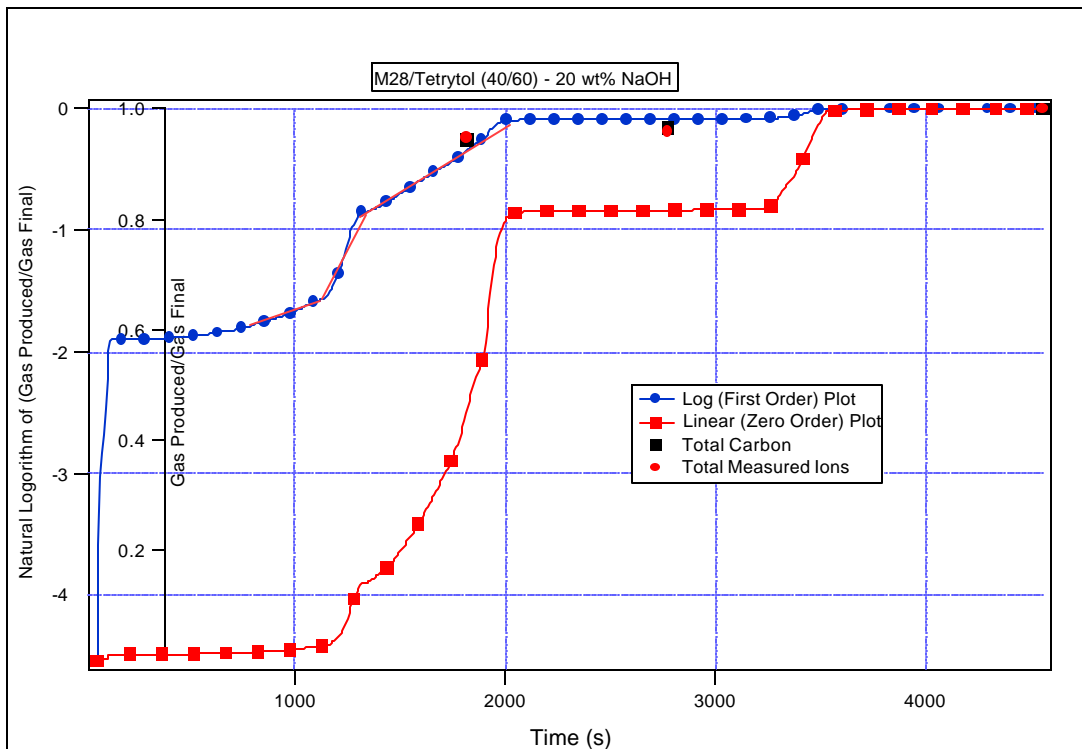
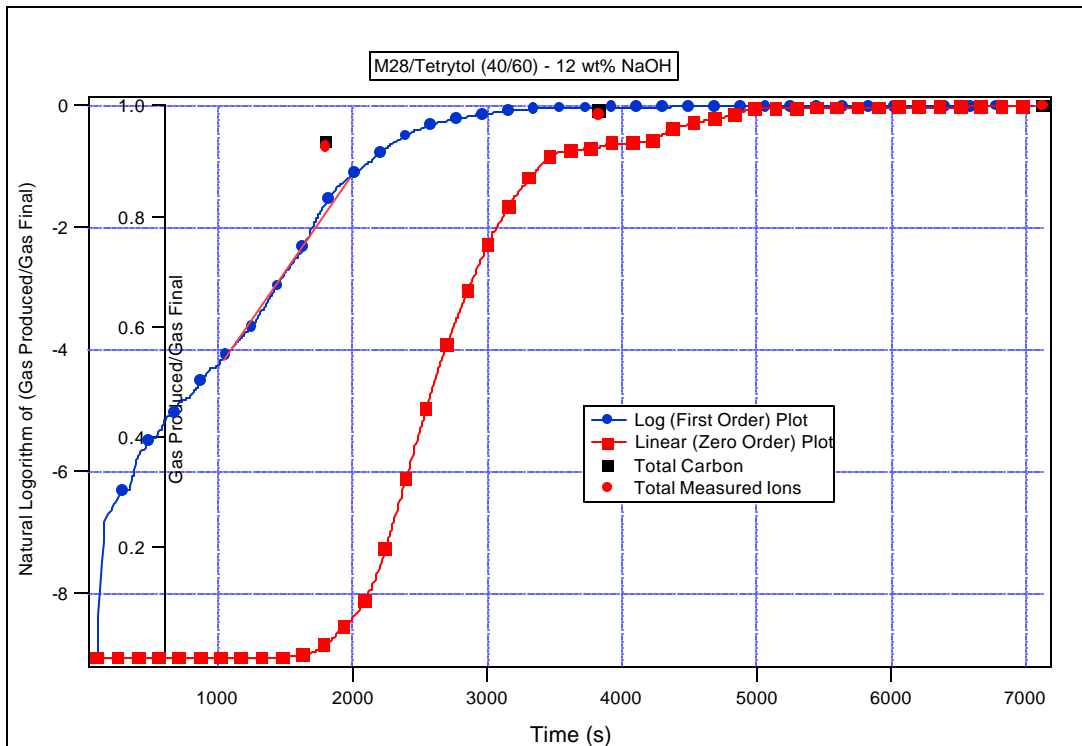




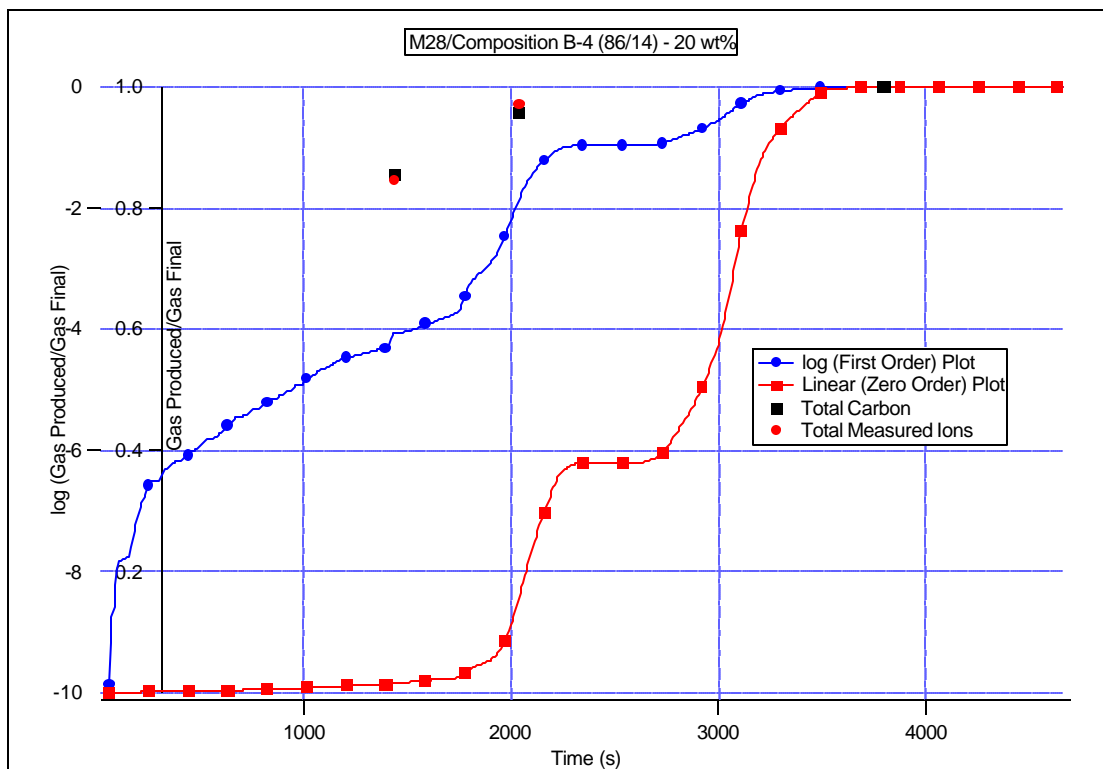
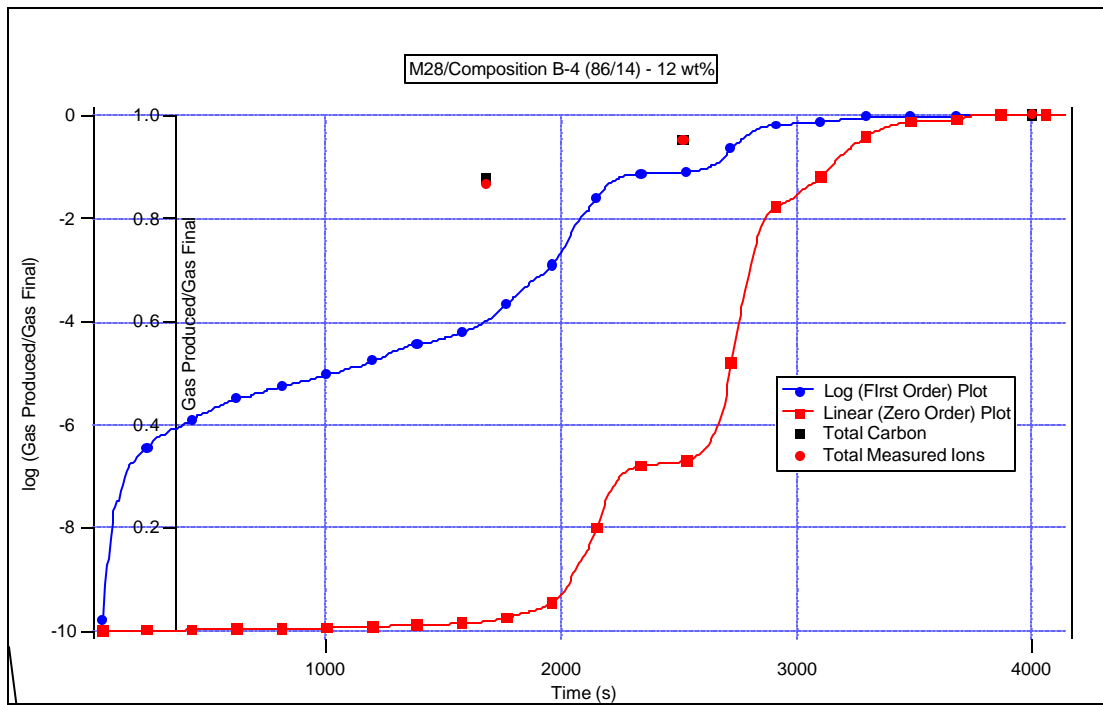
### 3.3.5 M28 Propellant



### 3.3.6 M28 Propellant/Tetrytol (40/60)



### 3.4.7 M28 Propellant with Composition B-4



### 3.4 TABULAR DATA

#### 3.4.1 Composition B

##### 12 wt% NaOH – Total Organic Carbon<sup>+</sup>

SAMPLE TIME (S)	TOTAL ORGANIC CARBON (PPM)	TOTAL INORGANIC CARBON (PPM)
1903.8	4437	11597*
4560	13328	1218
6480	14503	1427

##### 12 wt% NaOH – Ion Chromatography

SAMPLE TIME (S)	ACETATE (PPM)	FORMATE (PPM)	NITRITE (PPM)	NITRATE (PPM)	OXALATE (PPM)	AMMONIA (PPM)
1903.8	<174	698.61	3115	31	549	848.8
4560	<174	23238	16217	43	1305	3488.2
6480	<174	30894	21811	91	1505	2882.7

<sup>+</sup>This data was obtained from Dale Counce from EES-6, LANL. \*This value is unusually large, may be an error.

##### DSC of Hydrolysate Solution – 12 wt% NaOH

The DSC results showed no exotherm and no explosive properties of the hydrolysate solution.

##### HPLC Analysis of Comp B-4 Solution – 12 wt% NaOH

The HPLC analysis performed on Comp B-4 hydrolysate generated no peaks consistent with TNT, RDX or picric acid. However, this does not mean that there is no picric acid or tetryl present, just that there are no clear indication that picric acid or tetryl are present.

##### Final Reaction Products – 12 wt% NaOH

NO <sub>2</sub> (g/g)	NO <sub>3</sub> (g/g)	HCO <sub>2</sub> (g/g)	C <sub>2</sub> H <sub>3</sub> O <sub>2</sub> (g/g)	NH <sub>3</sub> (scc/g)	N <sub>2</sub> (scc/g)	N <sub>2</sub> O (scc/g)	NO (scc/g)
0.22	0.0009	0.31	ND	73	109	109	0.33

Overall Gas Production Rate – 12 wt% NaOH (Using Gas Production Rate) = 3.3%/min

##### 20 wt% NaOH – Total Organic Carbon<sup>+</sup>

SAMPLE TIME (S)	TOTAL ORGANIC CARBON (PPM)	TOTAL INORGANIC CARBON (PPM)
1544	5542	1209
2949	3193	1044
3050	20010	2210

##### 20 wt% NaOH – Ion Chromatography

SAMPLE TIME (S)	ACETATE (PPM)	FORMATE (PPM)	NITRITE (PPM)	NITRATE (PPM)	OXALATE (PPM)	AMMONIA (PPM)
1544	<174	7874	5899	36	421	2069.2
2949	<174	3184	3219	41	412	707.3
3050	<174	33956	21811	82	2567	1920.4

+This data was obtained from Dale Counce from EES-6, LANL

#### DSC of Hydrolysate Solution – 20 wt% NaOH

The DSC results showed no exotherm and no explosive properties of the hydrolysate solution.

#### HPLC Analysis of Comp B-4 Solution – 20 wt% NaOH

The HPLC analysis performed on Comp B-4 hydrolysate generated no peaks consistent with TNT, RDX or picric acid. However, this does not mean that there is no picric acid or tetryl present, just that there are no clear indication that picric acid or tetryl are present.

#### Final Reaction Products – 20 wt% NaOH

NO <sub>2</sub> (g/g)	NO <sub>3</sub> (g/g)	HCO <sub>2</sub> (g/g)	C <sub>2</sub> H <sub>3</sub> O <sub>2</sub> (g/g)	NH <sub>3</sub> (scc/g)	N <sub>2</sub> (scc/g)	N <sub>2</sub> O (scc/g)	NO (scc/g)
0.27	0.001	0.42	ND	77	218	129	0.22

#### Overall Gas Production Rate – 20 wt% NaOH (Using Gas Production Rate) = 7%/min

#### 3.4.2 Tetrytol

##### 12 wt% NaOH – Total Organic Carbon<sup>+</sup>

SAMPLE TIME (S)	TOTAL ORGANIC CARBON (PPM)	TOTAL INORGANIC CARBON (PPM)
1260	10370	2862
2200	27283	5507

##### 12 wt% NaOH – Ion Chromatography

SAMPLE TIME (S)	ACETATE (PPM)	FORMATE (PPM)	NITRITE (PPM)	NITRATE (PPM)	OXALATE (PPM)	AMMONIA (PPM)
1260	<174	2323	9892	119	3123	7355.3
2200	<174	5786	24325	224	7621	5149.9

+This data was obtained from Dale Counce from EES-6, LANL

#### DSC of Hydrolysate Solution – 12 wt% NaOH

The DSC results showed no exotherm and no explosive properties of the hydrolysate solution.

### HPLC Analysis of Tetrytol Solution – 12 wt% NaOH

The HPLC analysis performed on tetrytol hydrolysate generated in a small scale flask generate peaks consistent with TNT. There were no peaks consistent with tetryl or picric acid. However, this does not mean that there is no picric acid or tetryl present, just that there are no clear indication that picric acid or tetryl are present.

### Final Reaction Products – 12 wt% NaOH

NO <sub>2</sub> (g/g)	NO <sub>3</sub> (g/g)	HCO <sub>2</sub> (g/g)	C <sub>2</sub> H <sub>3</sub> O <sub>2</sub> (g/g)	NH <sub>3</sub> (scc/g)	N <sub>2</sub> (scc/g)	N <sub>2</sub> O (scc/g)	NO (scc/g)
0.24	0.002	0.058	ND	175	2.3	5.9	34

Overall Gas Production Rate – 12 wt% NaOH (Using Gas Production Rate) = 4.8%/min

### 20 wt% NaOH – Total Organic Carbon<sup>+</sup>

SAMPLE TIME (S)	TOTAL ORGANIC CARBON (PPM)	TOTAL INORGANIC CARBON (PPM)
1380	<87	2758
2040	9144	3497
3600	18296	6377

### 20 wt% NaOH – Ion Chromatography

SAMPLE TIME (S)	ACETATE (PPM)	FORMATE (PPM)	NITRITE (PPM)	NITRATE (PPM)	OXALTE (PPM)	AMMONIA (PPM)
1380	<174	145	788	10.8	164	119.5
2040	<174	2810	9962	157	3832	5044.3
3600	<175	6125	20332	261	8891	5735.9

<sup>+</sup>This data was obtained from Dale Counce from EES-6, LANL

### DSC of Hydrolysate Solution – 20 wt% NaOH

The DSC of the hydrolysate solution showed no exotherm and no explosive properties

### HPLC Analysis of Tetrytol Solution – 20 wt% NaOH

The HPLC analysis performed on tetrytol hydrolysate generated in a small scale flask generate peaks consistent with TNT. There were no peaks consistent with tetryl or picric acid. However, this does not mean that there is no picric acid or tetryl present, just that there are no clear indication that picric acid or tetryl are present.

### Final Reaction Products – 20 wt% NaOH

NO <sub>2</sub> (g/g)	NO <sub>3</sub> (g/g)	HCO <sub>2</sub> (g/g)	C <sub>2</sub> H <sub>3</sub> O <sub>2</sub> (g/g)	NH <sub>3</sub> (scc/g)	N <sub>2</sub> (scc/g)	N <sub>2</sub> O (scc/g)	NO (scc/g)
0.22	0.003	0.067	ND	226	51.7	10.0	37

Overall Gas Production Rate – 20 wt% NaOH (Using Gas Production Rate) = 9.3%/min

### 3.4.3 M1 Propellant

#### 12 wt% NaOH – Total Organic Carbon

SAMPLE TIME (S)	TOTAL ORGANIC CARBON (PPM)	STANDARD DEVIATION	TOTAL INORGANIC CARBON (PPM)	STANDARD DEVIATION
1910.4	1983.15	579.15	561.6	43.875
2825.4	9021.47	178.82	17.882	670.575
5200	19403	442.8	929.355	70.808

#### 12 wt% NaOH – Ion Chromatography

SAMPLE TIME (S)	ACETATE (PPM)	STANDARD DEVIATION	FORMATE (PPM)	STANDARD DEVIATION	NITRITE (PPM)	STANDARD DEVIATION
1910.4	5188.22	590.549	433.266	31.6641	1689.19	17.9119
2825.4	8989.43	656.393	3695.61	338.501	10971.4	106.223
5200	10608.8	129.255	2771.19	96.7174	24154.7	279.394

#### 12 wt% NaOH – Ion Chromatography

SAMPLE TIME (S)	NITRATE (PPM)	STANDARD DEVIATION	AMMONIA (PPM)	STANDARD DEVIATION
1910.4	15736.5	1413.41	4.19074	1.93995
2825.4	13005.4	1160.63	4.43556	0.917367
5200	9977.76	564.715	7.02091	2.53771

#### Final Reaction Products – 12 wt% NaOH

NO <sub>2</sub> (g/g)	NO <sub>3</sub> (g/g)	HCO <sub>2</sub> (g/g)	C <sub>2</sub> H <sub>3</sub> O <sub>2</sub> (g/g)	NH <sub>3</sub> (scc/g)	N <sub>2</sub> (scc/g)	N <sub>2</sub> O (scc/g)	NO (scc/g)
0.32	0.13	0.033	0.12	2.63	6.77	1.01	ND

Overall Gas Production Rate – 12 wt% NaOH (Using Gas Production Rate) = 3.6%/min

#### 20 wt% NaOH – Total Organic Carbon

SAMPLE TIME (S)	TOTAL ORGANIC CARBON (PPM)	STANDARD DEVIATION	TOTAL INORGANIC CARBON (PPM)	STANDARD DEVIATION
1206	3138.57	154.207	362.84	63.497
2096	19951.4	375.232	869.44	64.064
3825.94	20238.7	1900.48	891.693	99.077



20 wt% NaOH – Ion Chromatography

SAMPLE TIME (S)	ACETATE (PPM)	STANDARD DEVIATION	FORMATE (PPM)	STANDARD DEVIATION	NITRITE (PPM)	STANDARD DEVIATION
1206	5571.11	784.371	623.631	144.763	3953.44	150.994
2096	12446.7	153.909	2665.52	30.2674	25332	980.798
3825.94	10876	2029.79	3358.86	351.434	24656.7	784.97

20 wt% NaOH – Ion Chromatography

SAMPLE TIME (S)	NITRATE (PPM)	STANDARD DEVIATION	AMMONIA (PPM)	STANDARD DEVIATION
1206	3439.42	164.444	5.44529	0.615225
2096	9239.71	184.937	7.73732	1.86168
3825.94	8132.57	1004.64	11.7305	5.74777

DSC of Hydrolysate Solid Residue – 20 wt%

The thermal analysis of M1 propellant solid residue did not have a definitive melting point, but did have a low temperature exotherm at 30°C. It also has an exothermic decomposition starting at 165°C with melting at 181°C. The DSC reveals that 68.3% of the solid does not burn at 350°C and is probability sodium and hydroxide salts.

DSC of Hydrolysate Residue Tar – 20 wt%

The tar did not have any defined exothermic or endothermic reactions but may soften around 90C.

Final Reaction Products – 20 wt% NaOH

NO <sub>2</sub> (g/g)	NO <sub>3</sub> (g/g)	HCO <sub>2</sub> (g/g)	C <sub>2</sub> H <sub>3</sub> O <sub>2</sub> (g/g)	NH <sub>3</sub> (scc/g)	N <sub>2</sub> (scc/g)	N <sub>2</sub> O (scc/g)	NO (scc/g)
0.29	0.095	0.04	0.043	1.71	1.42	1.71	ND

Overall Gas Production Rate – 20 wt% NaOH (Using Gas Production Rate) = 7.3%/min

### 3.4.4 M8 Propellant

#### 12 wt% NaOH – Total Organic Carbon

SAMPLE TIME (S)	TOTAL ORGANIC CARBON (PPM)	STANDARD DEVIATION	TOTAL INORGANIC CARBON (PPM)	STANDARD DEVIATION
1520.4	3523.43	218.575	441.522	63.497
2035.2	14400.2	378.355	851.081	69.904
3665.94	16971.6	406.692	954.162	72.7353

#### 12 wt% NaOH – Ion Chromatography

SAMPLE TIME (S)	ACETATE (PPM)	STANDARD DEVIATION	FORMATE (PPM)	STANDARD DEVIATION	NITRITE (PPM)	STANDARD DEVIATION
1520.4	9535.33	255.017	1453.52	76.5013	7325.91	187.495
2035.2	10154.3	393.412	5119.01	202.091	33084.3	1063.7
3665.94	13142.5	194.135	6149.26	160.937	38977.9	542.472

#### 12 wt% NaOH – Ion Chromatography

SAMPLE TIME (S)	NITRATE (PPM)	STANDARD DEVIATION	AMMONIA (PPM)	STANDARD DEVIATION
1520.4	2604.69	114.795	25.9567	23.5653
2035.2	10045.1	279.397	15.6637	4.07164
3665.94	11698.9	69.3586	17.9933	0.508355

#### Final Reaction Products – 12 wt% NaOH

NO <sub>2</sub> (g/g)	NO <sub>3</sub> (g/g)	HCO <sub>2</sub> (g/g)	C <sub>2</sub> H <sub>3</sub> O <sub>2</sub> (g/g)	NH <sub>3</sub> (scc/g)	N <sub>2</sub> (scc/g)	N <sub>2</sub> O (scc/g)	NO (scc/g)
0.39	0.12	0.063	0.13	2.04	1.73	5.09	0.41

Overall Gas Production Rate – 12 wt% NaOH (Using Gas Production Rate) = 7.3%/min

#### 20 wt% NaOH – Total Organic Carbon

SAMPLE TIME (S)	TOTAL ORGANIC CARBON (PPM)	STANDARD DEVIATION	TOTAL INORGANIC CARBON (PPM)	STANDARD DEVIATION
1435.2	2883.88	555.229	546.942	174.027
2825.4	15976.3	1012.86	1020.78	71.217
5335.74	18648.4	275.672	1127.01	145.944

#### 20 wt% NaOH – Ion Chromatography

SAMPLE TIME (S)	ACETATE (PPM)	STANDARD DEVIATION	FORMATE (PPM)	STANDARD DEVIATION	NITRITE (PPM)	STANDARD DEVIATION
1435.2	20486.2	1003.37	1453.52	76.5013	7325.91	187.495
2825.4	19561.6	958.091	9386.8	415.432	47029.6	1676.98
5335.74	20043.7	981.701	9618.12	425.67	48188.5	1718.3

20 wt% NaOH – Ion Chromatography

SAMPLE TIME (S)	NITRATE (PPM)	STANDARD DEVIATION	AMMONIA (PPM)	STANDARD DEVIATION
1435.2	2604.69	114.795	7.68634	2.43619
2825.4	27128.4	1183.74	20.4976	2.6388
5335.74	27796.9	1212.92	23.8973	0.575943

DSC of Hydrolysate Solid Residue – 20 wt%

The thermal analysis of M8 propellant solid residue had a melting point at 58°C with a non-reversing exotherm associated with it. There were very weak exotherms at 52°C and 250°C. The DSC reveals that 72.6% of the solid does not burn at 350°C and is probability sodium and hydroxide salts.

Final Reaction Products – 20 wt% NaOH

NO <sub>2</sub> (g/g)	NO <sub>3</sub> (g/g)	HCO <sub>2</sub> (g/g)	C <sub>2</sub> H <sub>3</sub> O <sub>2</sub> (g/g)	NH <sub>3</sub> (scc/g)	N <sub>2</sub> (scc/g)	N <sub>2</sub> O (scc/g)	NO (scc/g)
0.48	0.28	0.093	0.05	16.3	7.83	16.3	1.8

Overall Gas Production Rate – 20 wt% NaOH (Using Gas Production Rate) = 9.5%/min

M28 Propellant

12 wt% NaOH – Total Organic Carbon

SAMPLE TIME (S)	TOTAL ORGANIC CARBON (PPM)	STANDARD DEVIATION	TOTAL INORGANIC CARBON (PPM)	STANDARD DEVIATION
1620	17018.7	1209.9	215.462	174.027
5446.2	19063.7	552.695	1266.95	59.521

12 wt% NaOH – Ion Chromatography

SAMPLE TIME (S)	ACETATE (PPM)	STANDARD DEVIATION	FORMATE (PPM)	STANDARD DEVIATION	NITRITE (PPM)	STANDARD DEVIATION
1620	17249.2	370.509	6842.45	249.511	31739.5	175.099
5446.2	17343.7	370.229	6855.75	200.616	31799.6	202.578

12 wt% NaOH – Ion Chromatography

SAMPLE TIME (S)	NITRATE (PPM)	STANDARD DEVIATION	AMMONIA (PPM)	STANDARD DEVIATION
1620	19156.7	40.1215	17.4535	3.58958
5446.2	18669.8	66.7823	19.3367	1.25182

DSC of Hydrolysate Solid Residue – 12 wt%

The thermal analysis of M28 propellant solid residue had a melting point at 68°C with a non-reversing exotherm associated with it. The weak exotherm could be the

crystallization of amorphous material, which then melts, called a cold crystallization. At 246°C a moderate exothermic decomposition begins. The DSC reveals that 72.5% of the solid does not burn at 350°C and is probability sodium and hydroxide salts.

Final Reaction Products – 12 wt% NaOH

NO <sub>2</sub> (g/g)	NO <sub>3</sub> (g/g)	HCO <sub>2</sub> (g/g)	C <sub>2</sub> H <sub>3</sub> O <sub>2</sub> (g/g)	NH <sub>3</sub> (scc/g)	N <sub>2</sub> (scc/g)	N <sub>2</sub> O (scc/g)	NO (scc/g)
0.51	0.30	0.11	0.28	5.30	5.30	4.27	0.058

Overall Gas Production Rate – 12 wt% NaOH (Using Gas Production Rate) = 2.9%/min

20 wt% NaOH – Total Organic Carbon

SAMPLE TIME (S)	TOTAL ORGANIC CARBON (PPM)	STANDARD DEVIATION	TOTAL INORGANIC CARBON (PPM)	STANDARD DEVIATION
1820.4	6699.22	729.876	585.639	69.512
2777.4	14575.1	781.74	967.62	112.918
7165.03	22889.7	1783.5	2122.8	143.55

20 wt% NaOH – Ion Chromatography

SAMPLE TIME (S)	ACETATE (PPM)	STANDARD DEVIATION	FORMATE (PPM)	STANDARD DEVIATION	NITRITE (PPM)	STANDARD DEVIATION
1820.4	15600.4	813.531	6111.26	193.176	28673.7	391.608
2777.4	13716.6	165.851	5092.17	190.239	22294.1	226.017
7165.03	19641.2	329.503	7488.06	185.003	35453.4	298.992

20 wt% NaOH – Ion Chromatography

SAMPLE TIME (S)	NITRATE (PPM)	STANDARD DEVIATION	AMMONIA (PPM)	STANDARD DEVIATION
1820.4	18359.1	339.548	23.0543	4.90947
2777.4	14179.9	92.7665	21.1457	7.32507
7165.03	22688.6	113.008	26.0617	9.66562

DSC of Solid Residue – 20 wt%

The thermal analysis of M28 propellant solid residue had a melting point at 74°C with a non-reversing exotherm associated with it. The DSC reveals that 62.6% of the solid does not burn at 350°C and is probability sodium and hydroxide salts.

### Final Reaction Products – 20 wt% NaOH

<b>NO<sub>2</sub> (g/g)</b>	<b>NO<sub>3</sub> (g/g)</b>	<b>HCO<sub>2</sub> (g/g)</b>	<b>C<sub>2</sub>H<sub>3</sub>O<sub>2</sub> (g/g)</b>	<b>NH<sub>3</sub> (scc/g)</b>	<b>N<sub>2</sub> (scc/g)</b>	<b>N<sub>2</sub>O (scc/g)</b>	<b>NO (scc/g)</b>
0.56	0.39	0.12	0.31	3.57	1.50	3.34	0.035

Overall Gas Production Rate – 20 wt% NaOH (Using Gas Production Rate) = 3.7%/min

#### 3.4.5 M28 Propellant with Tetrytol (40% M28, 60% Tetrytol)

##### 12 wt% NaOH – Total Organic Carbon

<b>SAMPLE TIME (S)</b>	<b>TOTAL ORGANIC CARBON (PPM)</b>	<b>STANDARD DEVIATION</b>	<b>TOTAL INORGANIC CARBON (PPM)</b>	<b>STANDARD DEVIATION</b>
1803	4628.99	746.064	1051.27	84.78
3822	16354.1	955.65	1869.75	24.93
7121.37	19676.9	1182.3	2854.41	126.675

##### 12 wt% NaOH – Ion Chromatography

<b>SAMPLE TIME (S)</b>	<b>ACETATE (PPM)</b>	<b>STANDARD DEVIATION</b>	<b>FORMATE (PPM)</b>	<b>STANDARD DEVIATION</b>	<b>NITRITE (PPM)</b>	<b>STANDARD DEVIATION</b>
1803	2788	290	0	0	7309.62	292
3822	9431	601	6535	230	23293	863
7121.37	11397	741	10260	216	33684	632

##### 12 wt% NaOH – Ion Chromatography

<b>SAMPLE TIME (S)</b>	<b>NITRATE (PPM)</b>	<b>STANDARD DEVIATION</b>	<b>AMMONIA (PPM)</b>	<b>STANDARD DEVIATION</b>
1803	3023	83	52.5017	23.5454
3822	6082	71	172.663	55.2899
7121.37	8305	256	250.082	9.32278

### Final Reaction Products – 12 wt% NaOH

<b>NO<sub>2</sub> (g/g)</b>	<b>NO<sub>3</sub> (g/g)</b>	<b>HCO<sub>2</sub> (g/g)</b>	<b>C<sub>2</sub>H<sub>3</sub>O<sub>2</sub> (g/g)</b>	<b>NH<sub>3</sub> (scc/g)</b>	<b>N<sub>2</sub> (scc/g)</b>	<b>N<sub>2</sub>O (scc/g)</b>	<b>NO (scc/g)</b>
0.39	0.095	0.099	0.16	9.72	4.46	1.59	2.78

Overall Gas Production Rate – 12 wt% NaOH (Using Gas Production Rate) = 4.5%/min

20 wt% NaOH – Total Organic Carbon

SAMPLE TIME (S)	TOTAL ORGANIC CARBON (PPM)	STANDARD DEVIATION	TOTAL INORGANIC CARBON (PPM)	STANDARD DEVIATION
1820.4	10920	2066.4	2032.8	65.352
2777.4	13851.6	378	2536.8	378
4557.5	21357	2975.7	2108.5	1028.74

12 wt% NaOH – Ion Chromatography

SAMPLE TIME (S)	ACETATE (PPM)	STANDARD DEVIATION	FORMATE (PPM)	STANDARD DEVIATION	NITRITE (PPM)	STANDARD DEVIATION
1820.4	8979	158	6433	239	20759	485
2777.4	8548	516	7306	201	22671	464
4557.5	10992	1094	12285	856	37243	380

12 wt% NaOH – Ion Chromatography

SAMPLE TIME (S)	NITRATE (PPM)	STANDARD DEVIATION	AMMONIA (PPM)	STANDARD DEVIATION
1820.4	3867	133	98.7443	18.3495
2777.4	5503	314	196.073	4.35618
4557.5	8761	213	466.207	48.82

DSC of Hydrolysate Solid Residue – 20 wt%

The thermal analysis of M8 propellant solid residue had a melting point at 68°C with a non-reversing exotherm associated with it. There was a broad exothermic decomposition at 130°C. The DSC reveals that 78% of the solid does not burn at 350°C and is probability sodium and hydroxide salts.

Final Reaction Products – 20 wt% NaOH

NO <sub>2</sub> (g/g)	NO <sub>3</sub> (g/g)	HCO <sub>2</sub> (g/g)	C <sub>2</sub> H <sub>3</sub> O <sub>2</sub> (g/g)	NH <sub>3</sub> (scc/g)	N <sub>2</sub> (scc/g)	N <sub>2</sub> O (scc/g)	NO (scc/g)
0.43	0.10	0.093	0.19	28.8	40.7	5.36	7.34

Overall Gas Production Rate – 20 wt% NaOH (Using Gas Production Rate) = 5.9%/min

### 3.4.6 M28 Propellant with Composition B-4 (86/14)

#### 12 wt% NaOH – Total Organic Carbon

SAMPLE TIME (S)	TOTAL ORGANIC CARBON (PPM)	TOTAL INORGANIC CARBON (PPM)
1680	782	286
2520	5559	409.8
4000	16634	1218

#### 12 wt% NaOH – Ion Chromatography<sup>+</sup>

SAMPLE TIME (S)	ACETATE (PPM)	FORMATE (PPM)	NITRITE (PPM)	NITRATE (PPM)	OXALATE (PPM)	AMMONIA (PPM)
1680	318	133	922	413	<87	29.3
2520	2636	974	5803	3576	508	47.2
4000	7169	3454	18044	9805	2010	302.4

<sup>+</sup>This data was obtained from Dale Counce from EES-6, LANL

#### Final Reaction Products – 12 wt% NaOH

NaNO <sub>2</sub> (g/g)	NaNO <sub>3</sub> (g/g)	NaHCO <sub>2</sub> (g/g)	NaC <sub>2</sub> H <sub>3</sub> O <sub>2</sub> (g/g)	NH <sub>3</sub> (scc/g)	N <sub>2</sub> (scc/g)	N <sub>2</sub> O (scc/g)	NO (scc/g)
0.23	0.12	0.044	0.091	10.7	28.6	24.0	ND

Overall Gas Production Rate – 12 wt% NaOH (Using Gas Production Rate) = 2%/min

#### 20 wt% NaOH – Total Organic Carbon

SAMPLE TIME (S)	TOTAL ORGANIC CARBON (PPM)	TOTAL INORGANIC CARBON (PPM)
1440	477	281
2041	7299	605
3800	18679	2306

#### 20 wt% NaOH – Ion Chromatography<sup>+</sup>

SAMPLE TIME (S)	ACETATE (PPM)	FORMATE (PPM)	NITRITE (PPM)	NITRATE (PPM)	OXALATE (PPM)	AMMONIA (PPM)
1440	381	70.5	413	261	<87	15.23346
2041	3437	1296	9492	5751	835	43.83272
3800	7778	4237	20897	12102	3315	253.0454

<sup>+</sup>This data was obtained from Dale Counce from EES-6, LANL

#### Final Reaction Products – 20 wt% NaOH

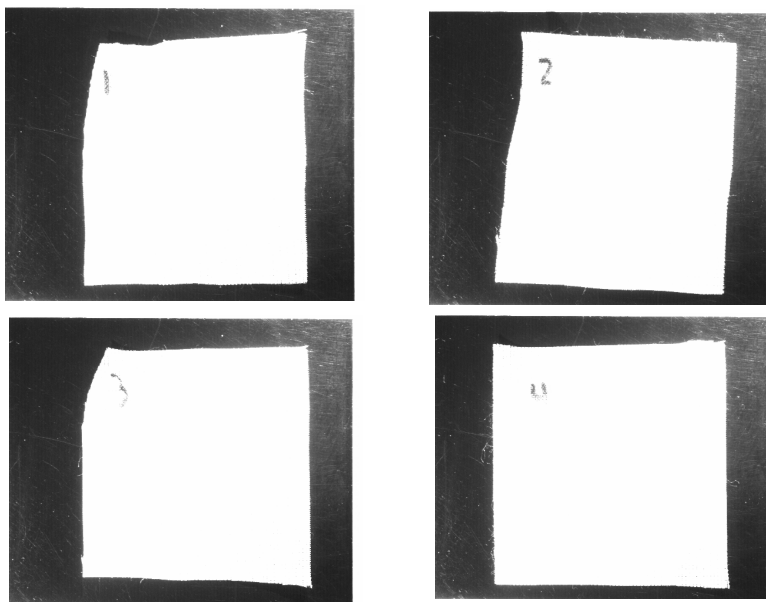
NaNO <sub>2</sub> (g/g)	NaNO <sub>3</sub> (g/g)	NaHCO <sub>2</sub> (g/g)	NaC <sub>2</sub> H <sub>3</sub> O <sub>2</sub> (g/g)	NH <sub>3</sub> (scc/g)	N <sub>2</sub> (scc/g)	N <sub>2</sub> O (scc/g)	NO (scc/g)
0.21	0.12	0.043	0.08	7.5	19.5	16.0	ND

Overall Gas Production Rate – 20 wt% NaOH (Using Gas Production Rate) = 1.7%/min

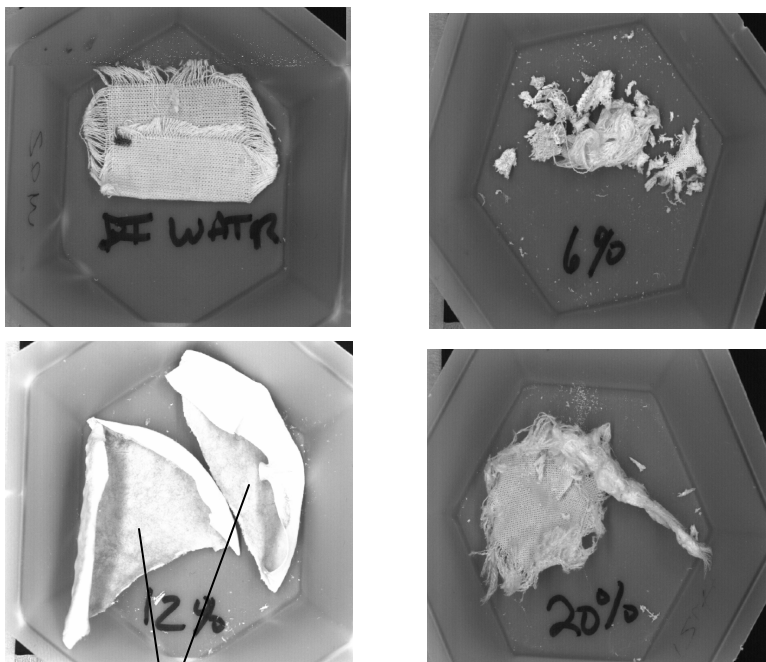
### 3.4.7 Experiments with the Rayon cloth

Two sets of four swatches of rayon cloth were exposed to 6, 12, and 20wt% NaOH at 93°C for 340 min. The swatches were photographed before and after the treatment with base. The pictures are shown below. It was not possible to determine the mass loss of the swatches due to a large amount of NaOH crystals adhering to the fibers when dried. Repeated rinsing of the swatches with water did not appear to improve the situation.

#### BEFORE



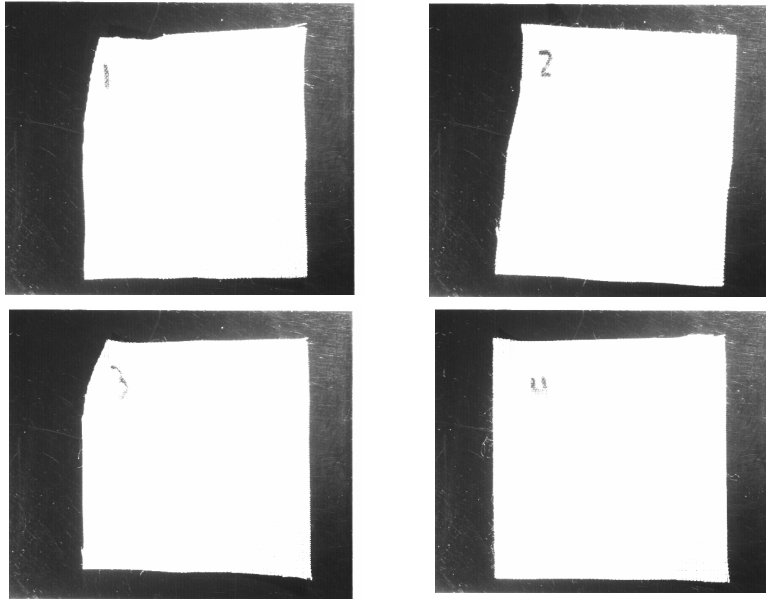
#### AFTER



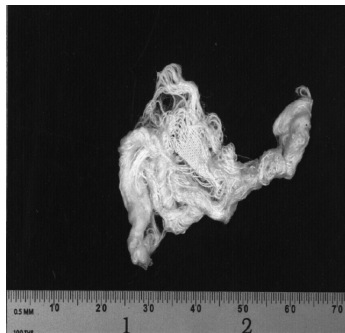
Strands on Filter Paper



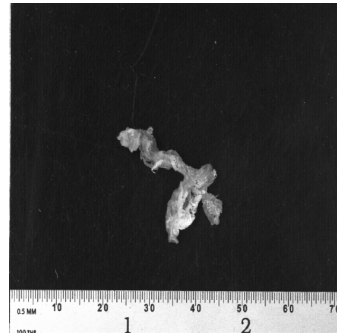
## BEFORE



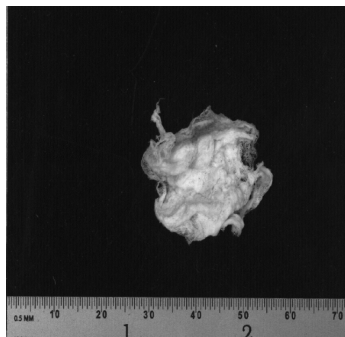
## AFTER



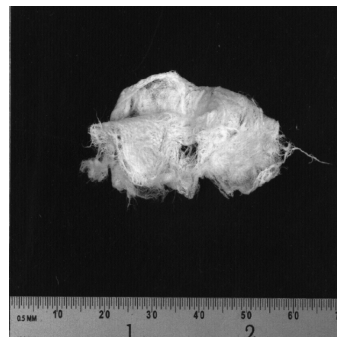
**Water**



**6% NaOH**



**12% NaOH**



**20 wt% NaOH**

### 3.5 DISCUSSION

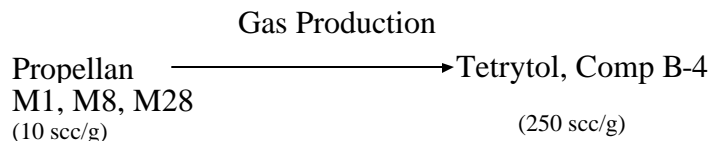
#### 3.5.1 Gas Reaction Products

The amount and type of gas produced during the base hydrolysis of the PMACWA explosives and propellants can be divided into three categories. The categories are, gas produced by explosive containing Composition B-4, gas produced by explosives containing Tetrytol, and gas produced by the propellants.

The principle gases produced by Composition B-4 were nitrous oxide ( $\text{N}_2\text{O}$ ), nitrogen ( $\text{N}_2$ ), and ammonia ( $\text{NH}_3$ ). These gas species are typical of the results found during previous hydrolysis experiments using this reactor with Composition B-3 and Composition B; with the exception of a much higher concentration of nitrogen gas in the product stream. This difference could be either from changes in experimental conditions (atmospheric with an argon sweep gas) or from experimental error derived from the difficulty in measuring nitrogen concentration using a mass spectrometer. Mass 28 (nitrogen's gas main peak) has a high amount of interference from  $\text{CO}$ , ( $\text{CO}_2$  breaks down to  $\text{CO}$  and  $\text{C}$  under the ion current) and  $\text{N}_2\text{O}$  (breaks down to  $\text{N}_2$  and  $\text{O}$  under the ion current). To further determine whether the additional nitrogen production is an actual result or a bi-product of using the mass spectrometer may be determined in the future using online gas chromatography to verify the gas species.

The principle gases produced by Tetrytol were nitrogen oxide ( $\text{NO}$ ), ammonia ( $\text{NH}_3$ ), nitrous oxide ( $\text{N}_2\text{O}$ ); and for 20 wt%  $\text{NaOH}$  nitrogen ( $\text{N}_2$ ) was also produced. In previous experiments in this reactor, the amount of nitrogen and nitrous oxide gas produced was minimal. These experiments also show that only a minimal amount of nitrous oxide and nitrogen were produced. There was significant amount nitrogen produced during the 20 wt%  $\text{NaOH}$  runs with Tetrytol. However previous experiments were not performed at base concentrations this high. A large amount of ammonia was produced during both runs with the majority of the gas being produced towards the end of the run. This may reflect continuing base reactions in the solution after the parent explosive compound has been degraded.

The propellants produced much less gas than Composition B-4 or Tetrytol. Although all four major gas products ( $\text{N}_2$ ,  $\text{N}_2\text{O}$ ,  $\text{NH}_3$ , and  $\text{NO}$ ) were detected, the quantity was minimal. A comparison between the different gases is shown in the figure below.



### 3.5.2 Aqueous Reaction Products

The concentrations and type of aqueous products produced during the base hydrolysis of the PMACWA explosives and propellants can be divided into two categories, those formed during the hydrolysis of Composition B-4 and Tetrytol, and those formed during the hydrolysis of the propellants. In the case of the melt castable explosives (Composition B-4 and Tetrytol), less than half of the nitrogen is contained in the aqueous phase. The aqueous nitrogen is in the form of nitrite with very little nitrate present. The vast majority of the carbon products are in the aqueous phase. Most of the carbon is organic with very little of it identified. The major carbon species identified are formate and oxalate. The overall nitrogen balance for the Composition B-4 and Tetrytol hydrolysis is much higher than previous experiments have yielded.

For propellants, the majority of the base hydrolysis products are aqueous in the form of nitrite, nitrate, acetate and formate. These ions account for nearly all the nitrogen in the propellant. The vast majority of the carbon products are also found in the aqueous phase. However, similar to the base hydrolysate of Composition B-4 and Tetrytol, very little of the carbon products have been identified. The major carbon species detected are acetate and formate.

Two different groups at Los Alamos (DX-2 and EES-6) performed the aqueous product analysis for Composition B-4 and Tetrytol. Except for the additional detection of oxalate and possibly glycolate in the EES-6 analysis (these compounds were not looked for by DX-2), the results were found to be very similar. The largest percent error between the two results was 16% (formate in Composition B-4, 12 wt%).

### 3.5.3 Gas Production Rate/ Reaction Rates

The gas production rate was used to estimate the overall reaction rates. The percent conversion was plotted on both a semi-log (first order in NaOH or explosive) and a linear (zero-order) graph. The semi-log plot showed some linear trends and gives support to using first order reaction mechanism for base hydrolysis. The literature usually lists a bimolecular (first order in both base and explosive) as the reaction mechanism for base hydrolysis. However, for solid-liquid reactor systems, mass transfer dominates. When mass transfer dominates, the rate is proportional to the explosive's specific surface area and the base concentration. The specific surface area of the explosive, and the base concentration, both decreases during the hydrolysis run. This would cause a decrease in the conversion rate. A complete model would need to include the change in particle size, explosive mass, base concentration, and diffusivities of both the explosive and OH<sup>-</sup> during the experimental run. For a first approximate the effective rate was assumed to be first order.

The effect of base concentration on hydrolysis rate for the explosives and propellants used in this study appears to be significant. This is very different that for the base hydrolysis of HMX. In HMX, the rate was shown to slow down at

base concentration above 1.5 M NaOH. The decrease in the rate was due to the lowering of the solubility of HMX caused by an increase in the ionic strength of the solution when high NaOH concentrations were used. This phenomenon does not appear to occur in the explosive and propellants used in this study. This difference may be due to the higher solubility of the explosives and propellants in water (compared to HMX).

A comparison of the overall reaction rates for the explosives and propellants studied in these experiments is shown in the table below.

<b>Explosive</b>	<b>Base Concentration (wt%)</b>	<b>Overall Reaction Rate (%/min)</b>
Composition B-4	12 wt%	3.3
	20 wt%	7.0
Tetrytol	12 wt%	4.8
	20 wt%	9.3
M1	12 wt%	3.6
	20 wt%	7.3
M8	12 wt%	7.3
	20 wt%	9.5
M28	12 wt%	2.9
	20 wt%	3.7
M28/Tetrytol	12 wt%	4.5
	20 wt%	5.9
M28/Composition B-4	12 wt%	2.0
	20 wt%	1.7

#### 3.5.4 Safety of the Base Hydrolysis Product

The DSC and HPLC result for all of the hydrolysate liquids and solid residues analyzed indicated no appreciable amounts of explosive molecules or explosive intermediates remaining. Specifically, picric acid was not found in either the Composition B-4 or Tetrytol hydrolysate. The DSC results showed no large exotherms for any of the base hydrolysate or solid residue. This result would indicate that there are little or no residual explosives in the hydrolysate liquid or solid residue. The tests performed on the hydrolysate show it to be safe to handle with the only intrinsic hazard being the high pH (13-14) of the final solution. .

#### 4.0 Design and Operating Suggestions for 2000-gal Reactor

The experiments performed in this study reflect the base hydrolysis of a few propellants and high explosive materials in a 2000 mL Parr reactor at a temperature of 93C and at atmospheric pressure. The rate and products are representative of the degradation of these explosive and propellant by base hydrolysis, but also reflect the stirrer rate, stirrer type, reactor type, and operational temperatures and pressures. To apply the reaction rate and product production rate to a different reactor, some design factors must be employed. For the base hydrolysis of bulk explosive and propellant, the reaction rate is dominated by the mass transfer rate. Therefore, the base hydrolysis reaction rate is related to stirring rate, stirrer size, reactor volume, and the diffusivity of the reactants. The following equations show the proper relationships.[1-8]

$$\begin{aligned} \text{Reaction Rate} &\propto (\text{Power} * \text{Viscosity})^{0.25} * \text{Sc}^{\frac{2}{3}} \\ \text{Power} &\propto \frac{P}{\text{Solution Mass}} \propto \frac{P_o * \text{RPM}^3 * (\text{Stirrer Diameter})^5}{\text{Solution Mass}} \\ \text{Power} &\propto \frac{(\text{Re}_{\text{impeller}})^{\gamma} * \text{RPM}^3 * (\text{Stirrer Diameter})^5}{\text{Solution Mass}} \\ \text{Re}_{\text{impeller}} &\propto \frac{(\text{Stirrer Diameter})^2 * \text{RPM}}{\text{Viscosity}} \\ \text{Sc} &\propto \frac{\text{Viscosity}}{\text{Diffusivity}} \\ \gamma &\equiv \text{Function of Stirrer Type and Reactor Configuration} \end{aligned}$$

The constant  $\gamma$ , is dependent upon the number of stirrers, stirrer shape, baffling and other factors. .

#### 5.0 Conclusions

Base hydrolysis was shown to be an effective method for degrading the explosives and propellants studied under the PMACWA program. The calculated overall reaction times for complete destruction of the energetic component in the explosives and propellants studied varied from 15 to 30 minutes at 93C. The major gas species were nitrogen, nitrous oxide, nitrogen oxide, and ammonia. The major aqueous species measured were ammonia, nitrite, nitrate, formate and acetate. The amount of gas produced during the base hydrolysis of explosives and propellants varied in volume from 10 SCC/gram for the propellant to 270 SCC/gram for Composition B-4 and Tetrytol.

The nitrogen contained in the melt castable explosives (Composition B-4 and Tetrytol) was split between the gas and liquid phase. In the case of the propellants, the majority of the nitrogen was found in the liquid phase. The overall nitrogen balance for the base hydrolysis gaseous and liquid products varied from 70% to 150%; this is not adequate to

claim that all the nitrogen species have been identified. No carbon balance was calculated because of the large amount of unknown carbon species in the base hydrolysate liquid.

Although the product distribution in the base hydrolysate is not entirely known, HPLC and DSC was performed on both the base hydrolysate liquid and solid residue generated during the experimental runs. For all the samples tested, there were no explosive compounds detected. Furthermore, there is no experimental evidence to support the formation of picrates during base hydrolysis. The base hydrolysis of explosives and propellants produces a safe, non-explosive product.

## 6.0 Acknowledgements

Los Alamos National Laboratory would like to acknowledge the PMACWA program for funding this research, specifically Scott Sussman, Bishara Elamasri and Peter Bonnet. Special thanks to Jose' Archuleta, Dale Counce, Gregg Sullivan, Ken Laintz, Sheldon Larson, Ray Flesner and John Sanchez for all their help.

## 7.0 Mass Transfer References

1. Bishop, R.L., *et al.*, *The Application of Gas Liquid Film Theory to Base Hydrolysis of HMX powder and HMX-based Plastic Bonded Explosives using Sodium Hydroxide*. Accepted for Publication in Ind. Eng. Chem. Res., 1999.
2. Kawase, Y. and M. Moo-Young, *Solid-Turbulent Fluid Heat and Mass Transfer: A Unified Model Based on the Energy Dissipation Rate Concept*. The Chemical Engineering Journal, 1987. 36: p. 31-40.
3. Kawase, Y. and M. Moo-Young, *Volumetric Mass Transfer Coefficients in Aerated Stirred Tank Reactors with Newtonian and Non-Newtonian Media*. Eng. Res. Des., 1988. 66: p. 284-288.
4. Kawase, Y. and M. Moo-Young, *Mass Transfer at a Free Surface In Stirred tank Bioreactors*. Trans. IChemE, 1990. 68(A): p. 189-194.
5. Treybal, R.E., *Mass-Transfer Operations*. 3 ed. Chemical Engineering Series. 1980, New York: McGraw-Hill. 522-533.
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8. Hikita, H. and S. Asai, *Gas Absorption with (m,n)-the Order Irreversible Chemical Reaction*. Int. Chem. Eng., 1964. 4(2): p. 332-340.

## **APPENDIX D**

**Final Report, Heat of Reaction for the Base Hydrolysis of Composition B, Tetrytol, M1, M8, and M28 Propellant, Los Alamos National Laboratory**

# Final Report

Heat of Reaction for the Base Hydrolysis of Composition B, Tetrytol, M1, M8, and M28  
Propellant

Robert Bishop and John Sanchez  
Los Alamos National Laboratory



*In order to further evaluate the use of alkaline hydrolysis for the first step in chemical weapons demilitarization, the hydrolysis of Composition B, Tetrytol, and M1, M8 and M28 propellant is currently being studied at a production scale (2000-gal batches) for the Program Manager for Assembled Chemical Weapons Activities (PMACWA) program. To support this study, 10-g sample of explosives and propellants were hydrolyzed in 12, 20, and 35-wt% NaOH at 90-110°C. These studies were designed to measure the heat of reaction. The heat of reaction was measured by using a simple differential thermal analysis technique using a four place hot plate/stirrer and a data-logging computer. The average and peak differential heats of reaction are presented in this report.*

## **1.0 Introduction**

The United States and several other countries are actively developing programs in the field of chemical weapon demilitarization. The PMACWA program has been tasked with investigating several different technologies as alternatives to incineration. Many of these technologies will use base hydrolysis as the first step. In order to investigate base hydrolysis at an industrial scale, a 2000-gal reactor has been constructed at the Holston ammunition plant (Operated by Royal Ordnance North America). To support the operation of the large-scale reactor, bench-scale studies to determine the heat of reaction are being performed. This report presents those results.

## **2.0 New Results under the PMAWA Studies**

### **2.1 EXPERIMENTAL SPECIFICS**

#### **2.1.1 Materials**

The explosives and propellants were provided through Picatinny Arsenal and the PMACWA program. The compounds studied were Comp B-4 (59.75% RDX, 39.75% TNT, and 0.5% Calcium Silicate), Tetrytol (70% tetryl and 30% TNT), M1 Propellant (85% nitrocellulose, 10% DNT, 5% dibutylphthalate, 1% diphenylamine), M8 propellant (52.15% nitrocellulose, 43% nitroglycerin, 1.25% potassium nitrate, 3% diethylphthalate, 0.60% ethyl centralite), and M28 propellant (60% nitrocellulose, 23.8% nitroglycerin, 9.9% triacetin, 2.3% dimethylphthalate, and 2.2% lead stearate). The Comp B-4 was provided as large flakes, the Tetrytol was in small to medium chunks, the M1 propellant was in 30-35 g sheets, and the M1 and M8 propellants were small grains.

The sodium hydroxide was made using dry-pellets of NaOH (over 98.5% pure) and house de-ionized water.

#### **2.1.2 Reactor System**

The reactor system used for these studies was a four place hot plate/stirrer with feed back temperature control. Four 250 ml Erlenmeyer flasks were used for each experimental run. Three of those flasks contained 10 g of explosive or propellant and 100 g of 12, 20 or 35 wt% NaOH. The remaining flask contained just the base solution with no propellant or explosive. Each flask contained a thermocouple. The temperature readings were recorded using Labview™ (National Instruments) on a Macintosh™ computer.

#### 2.1.3 Thermocouples

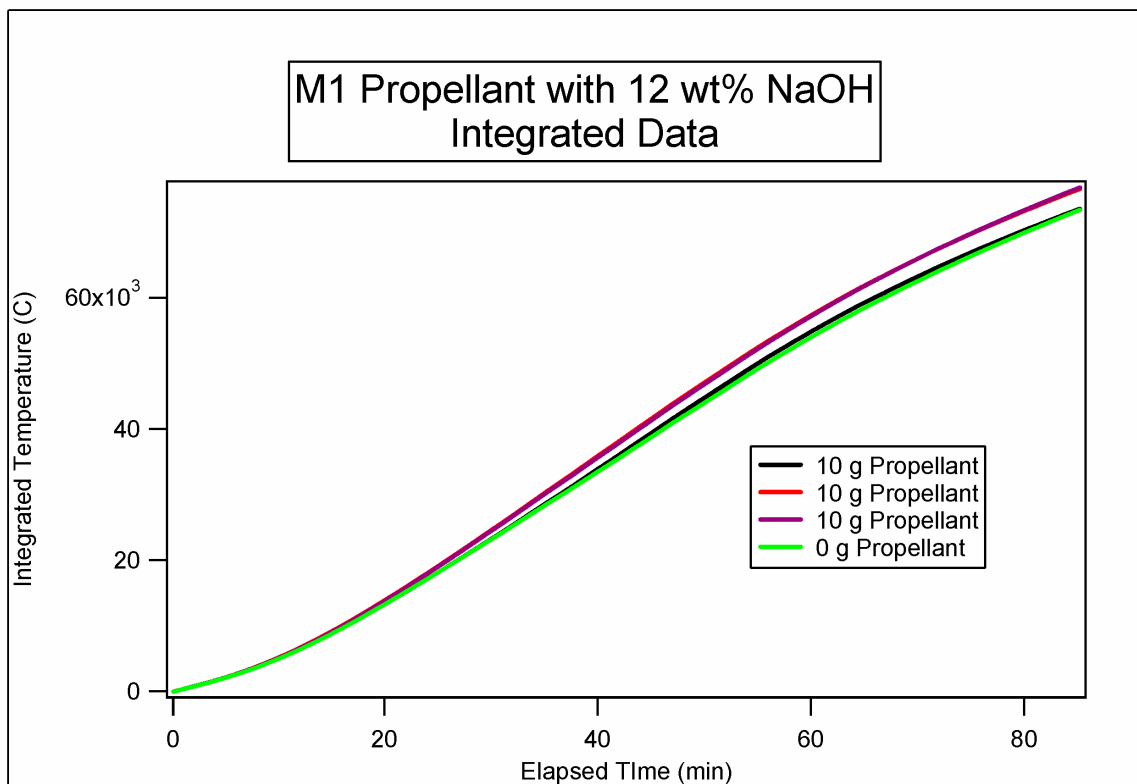
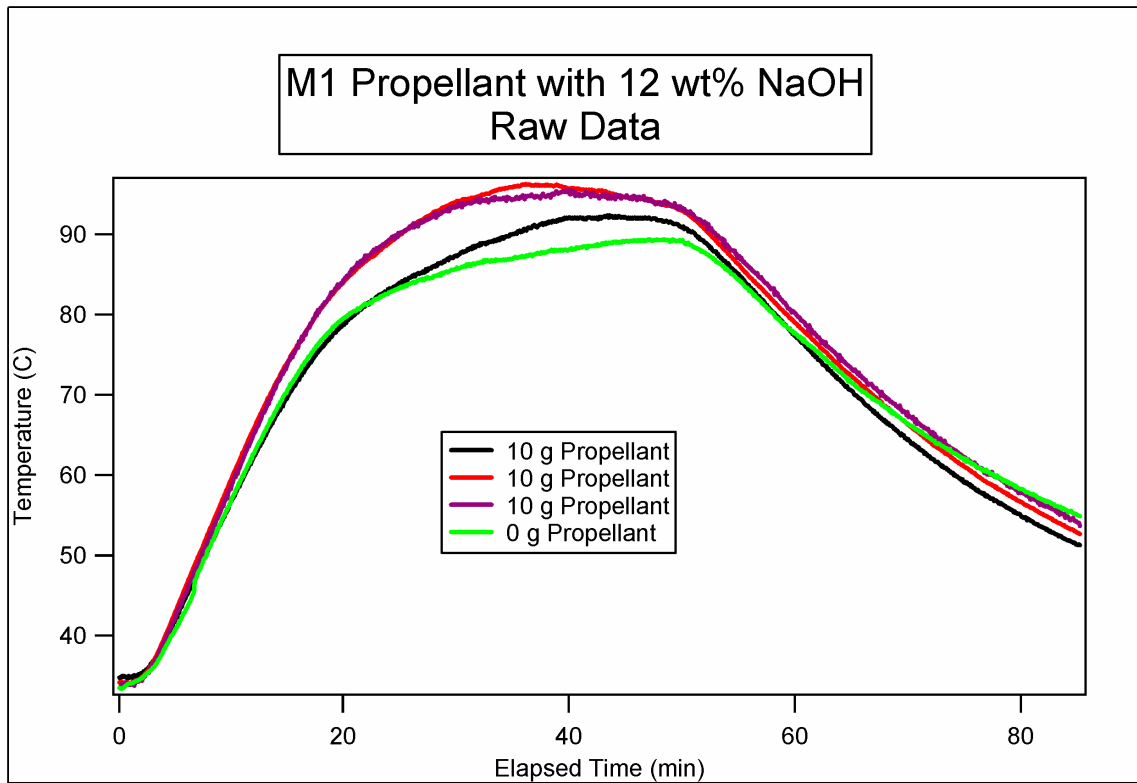
The thermocouples were Omega type K thermocouples. This type is well suited for the temperature ranges studied (20°C – 160°C).

### **3.0 Results**

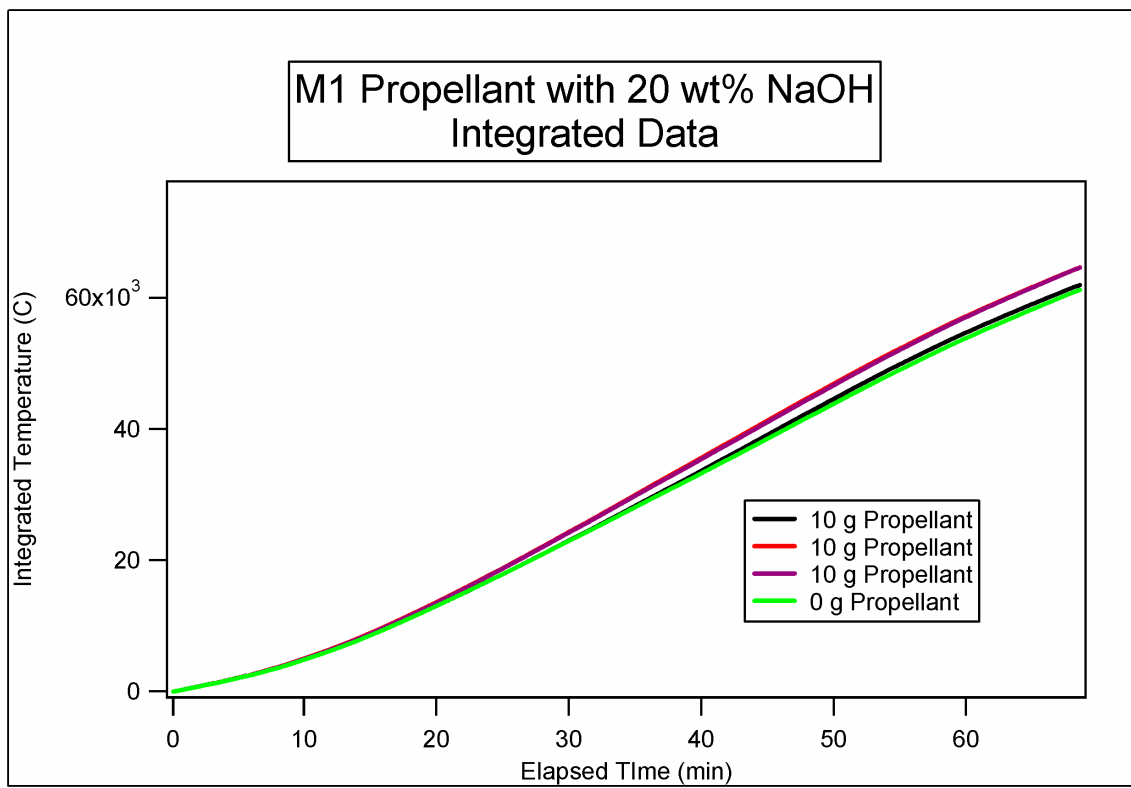
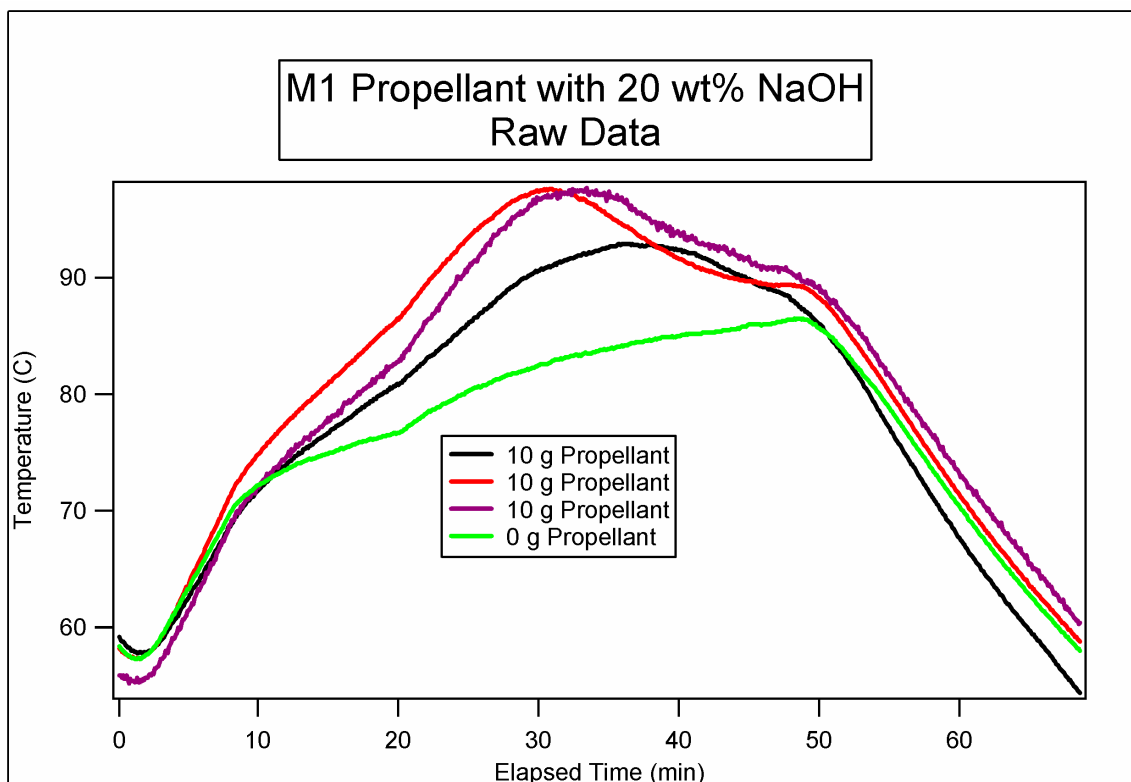
#### *3.1 TIME-TEMPERATURE PLOTS*

For each experimental run, two plots are presented. The first plot contains the raw time-temperature curves and the second contains the integration of those curves.

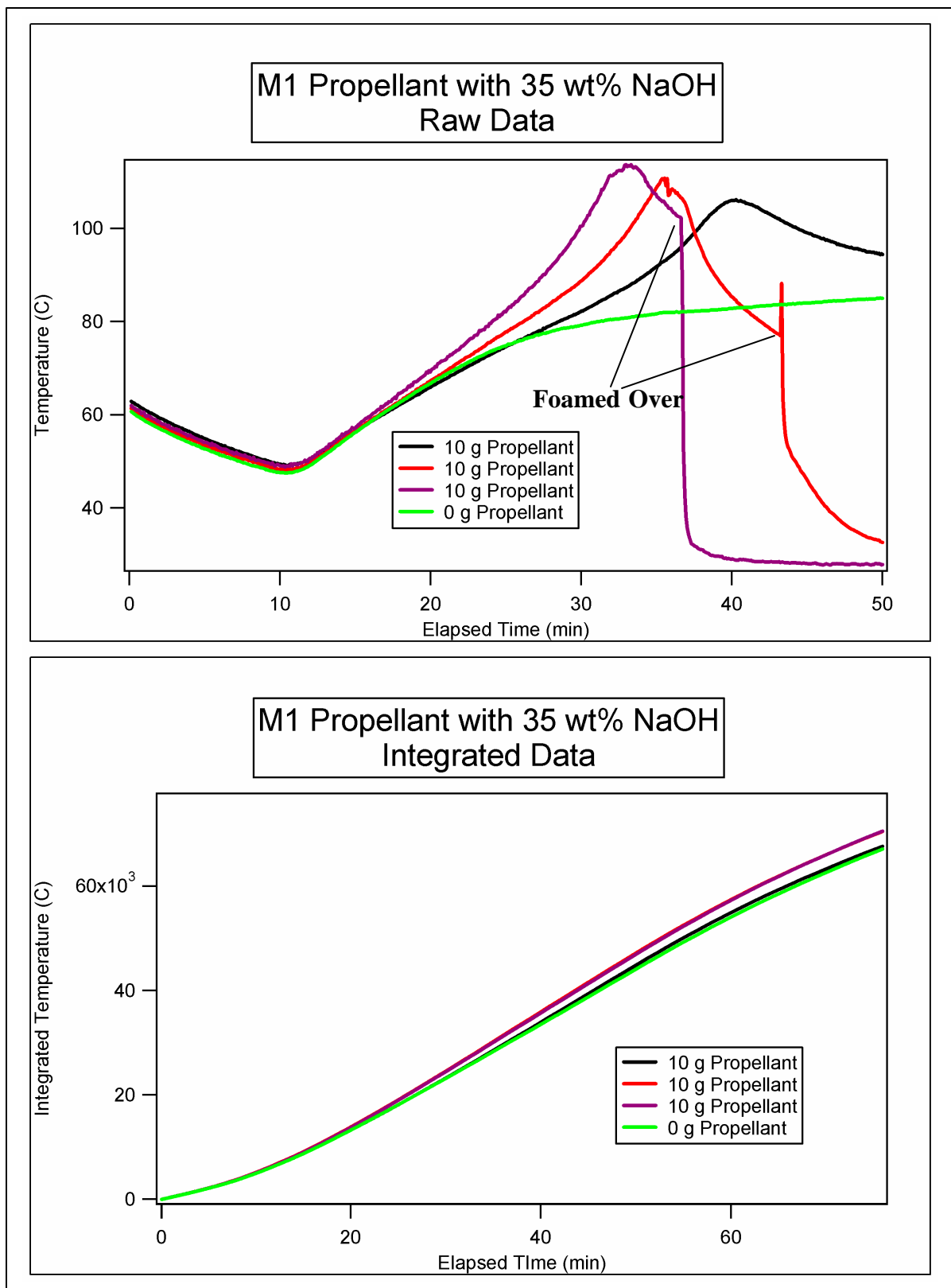
3.1.1 M1 Propellant  
3.1.1.1 12 wt% NaOH, 10 g Propellant/100g Base



3.1.1.2 20 wt% NaOH, 10 g Propellant/100g Base

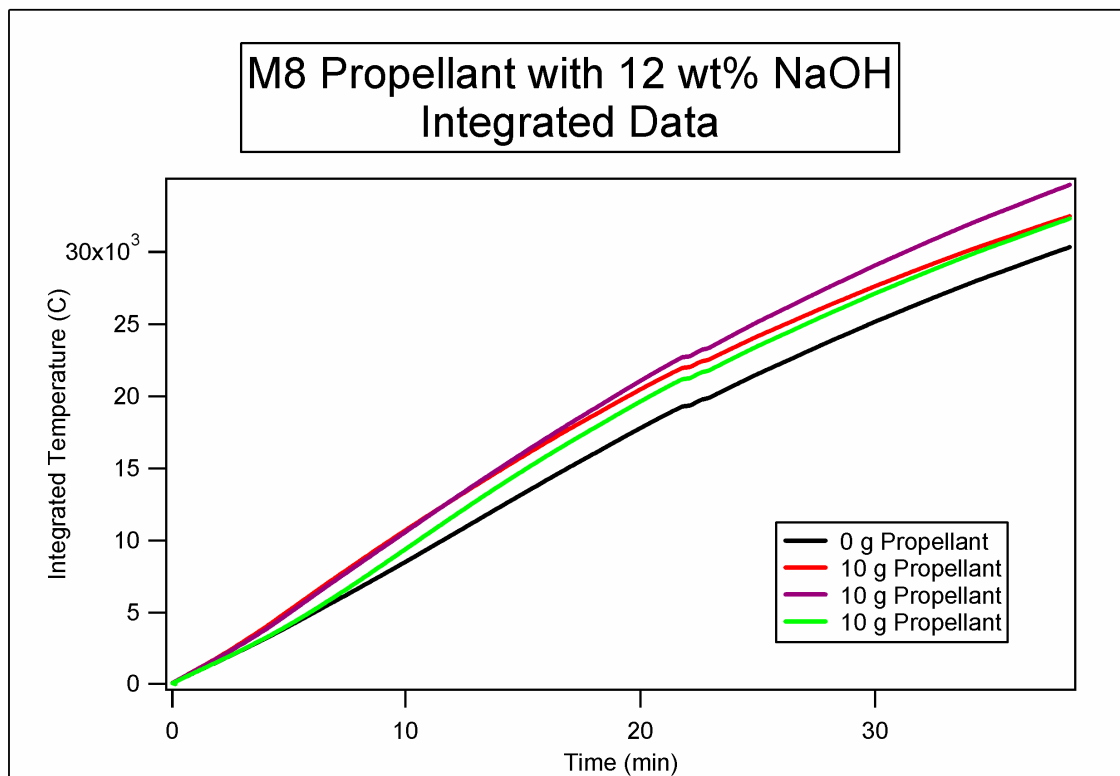
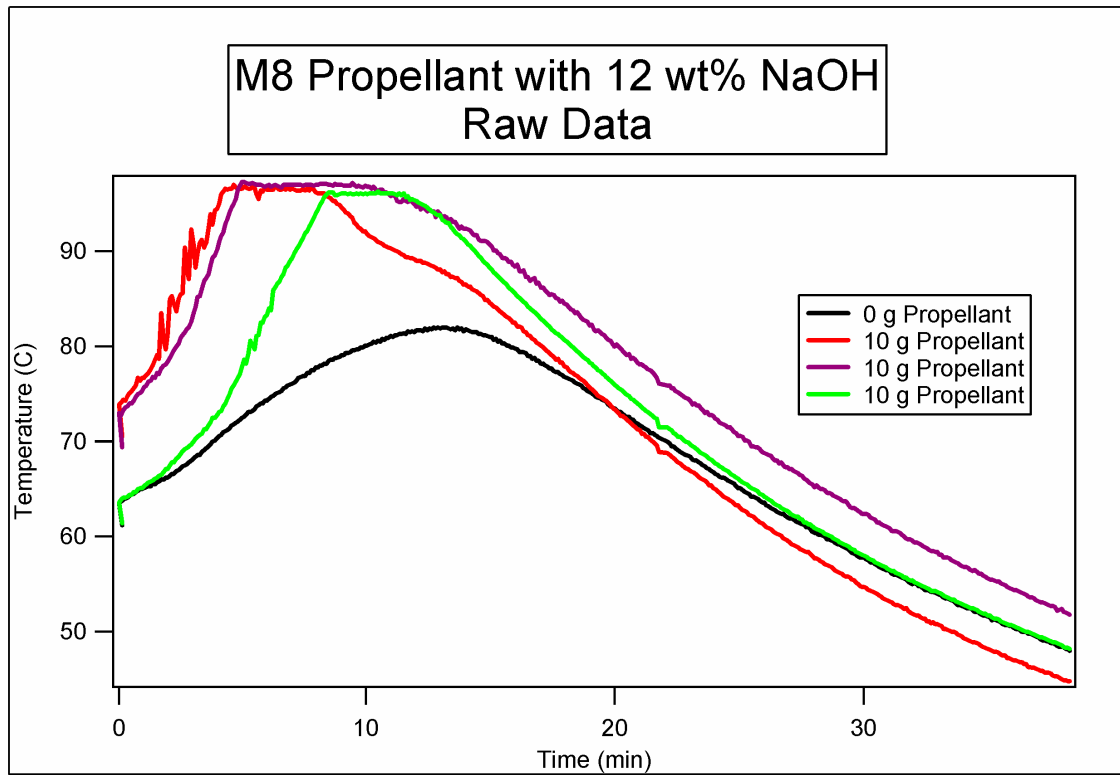


3.1.1.3 35 wt% NaOH, 10 g Propellant/100g Base

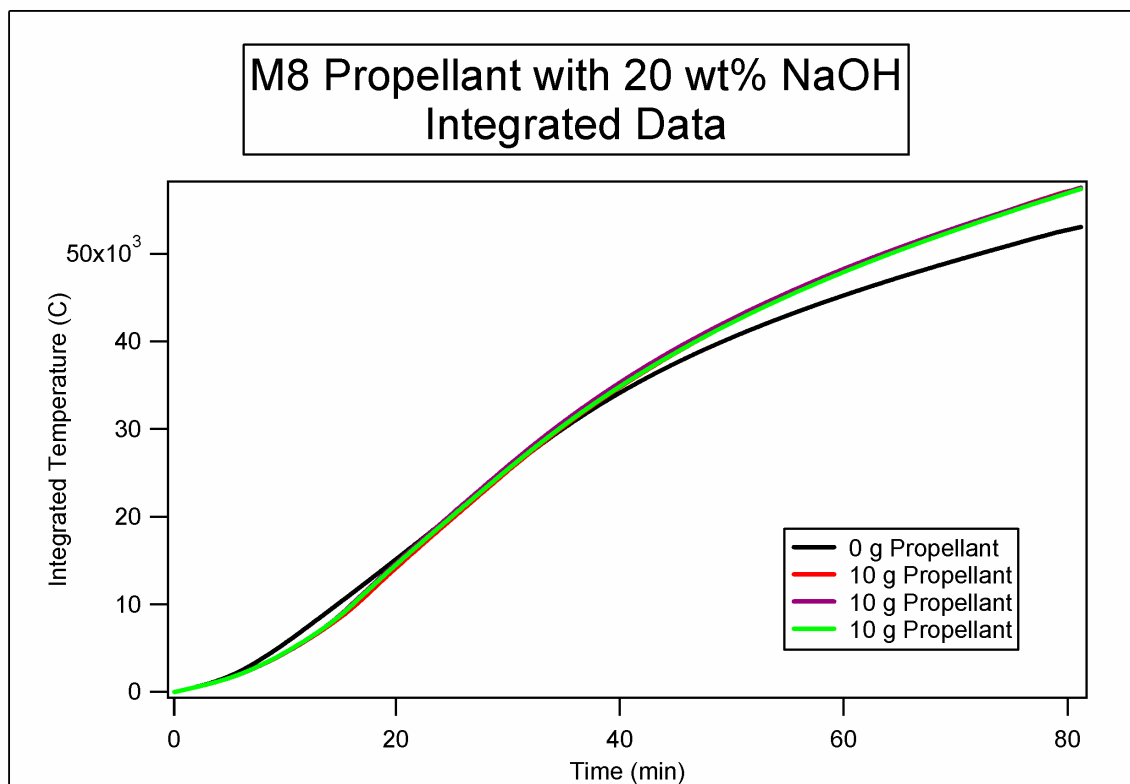
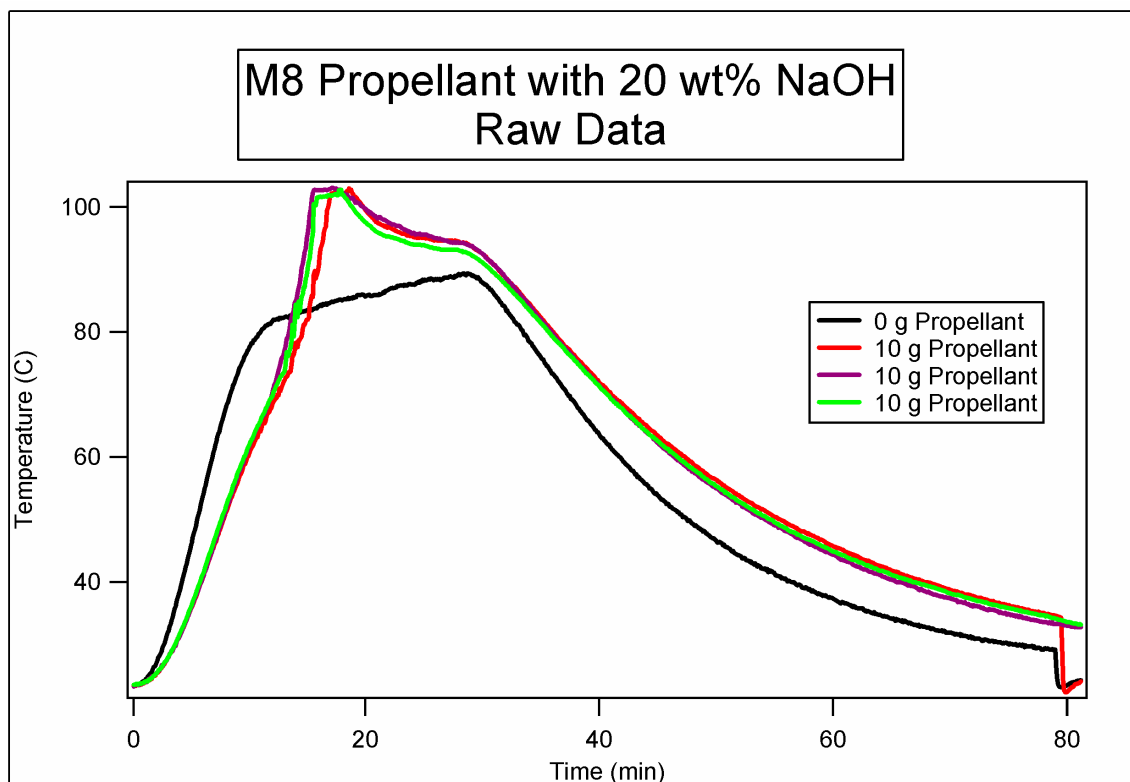


### 3.1.2 M8 Propellant

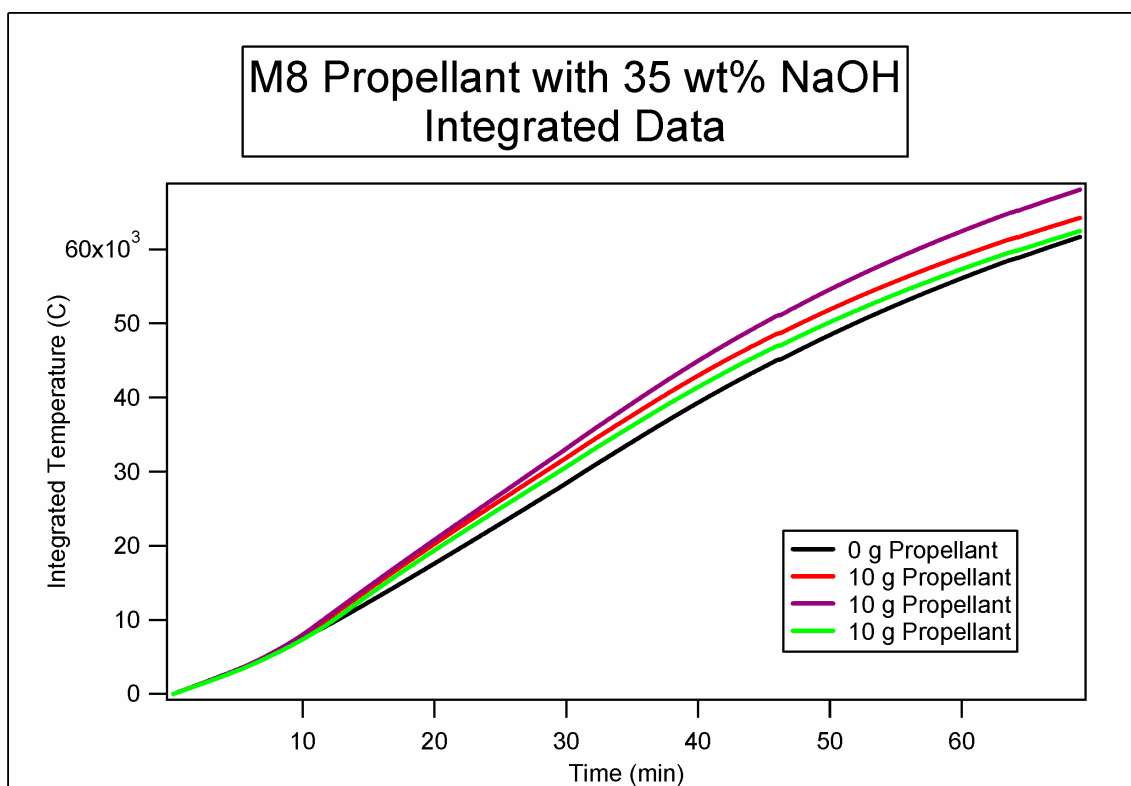
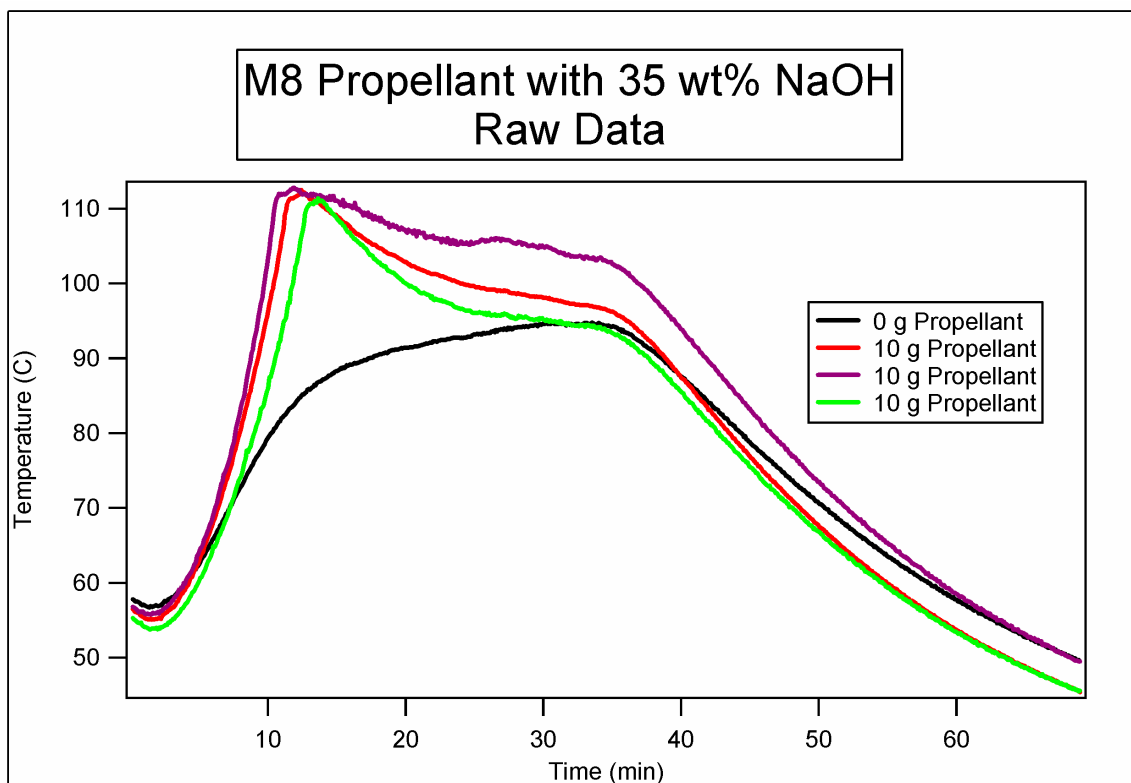
#### 3.1.2.1 12 wt% NaOH, 10 g Propellant/100 g Base



3.1.2.2 20 wt% NaOH, 10 g Propellant/100 g Base



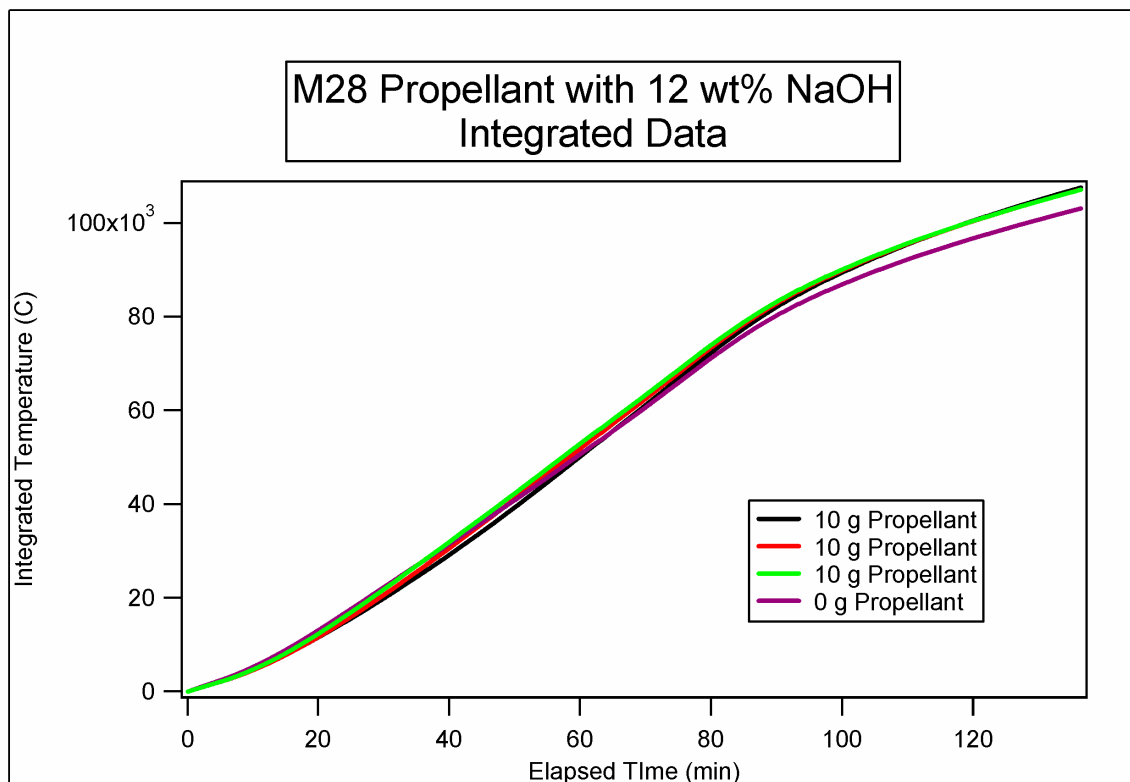
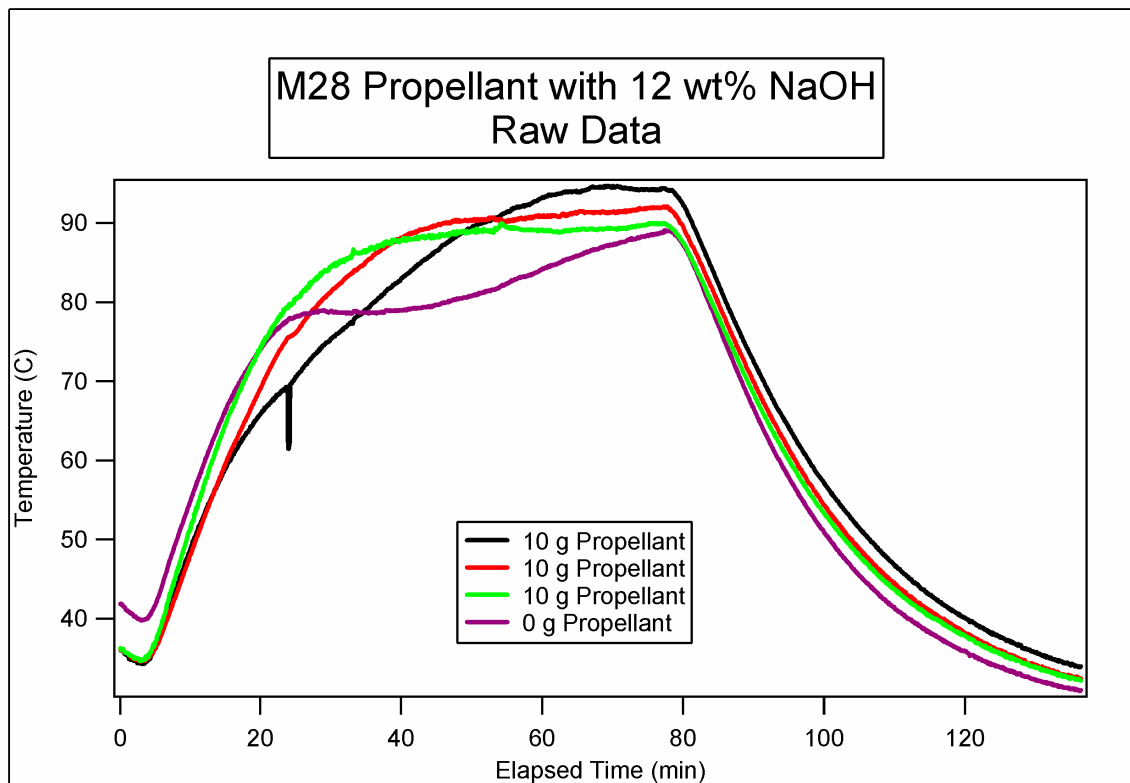
### 3.1.2.3 35 wt% NaOH, 10 g Propellant/100 g Base



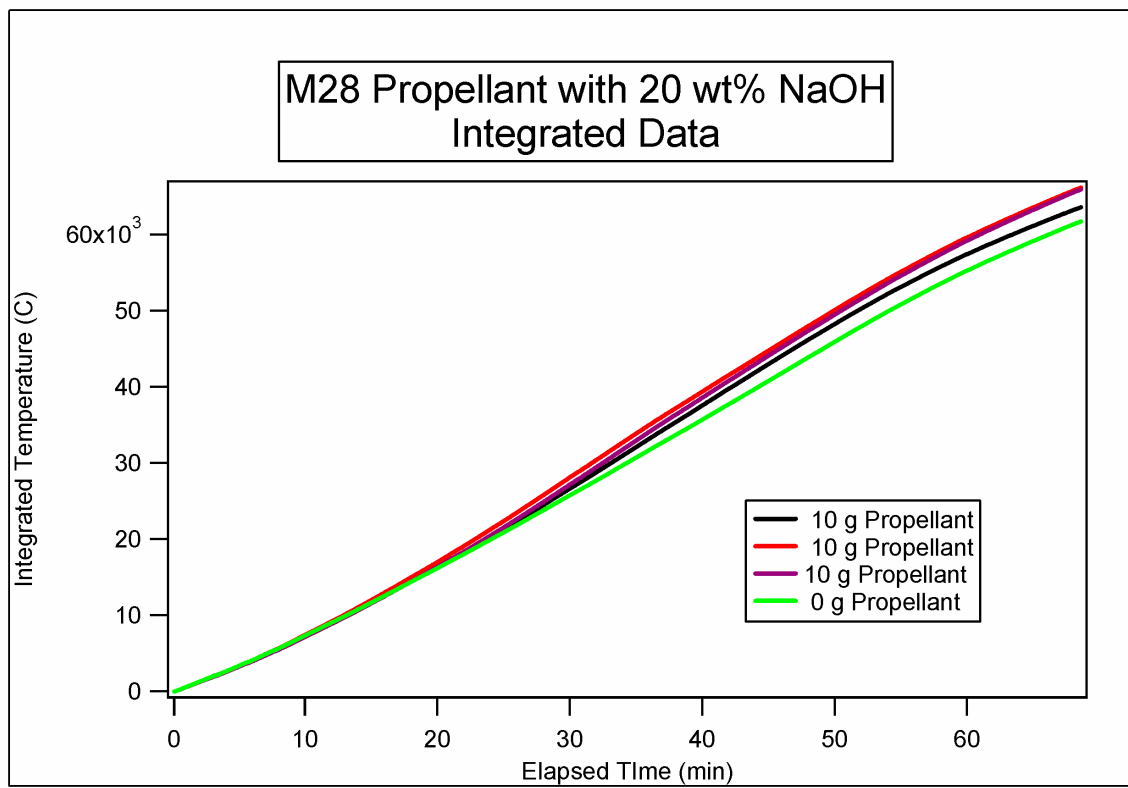
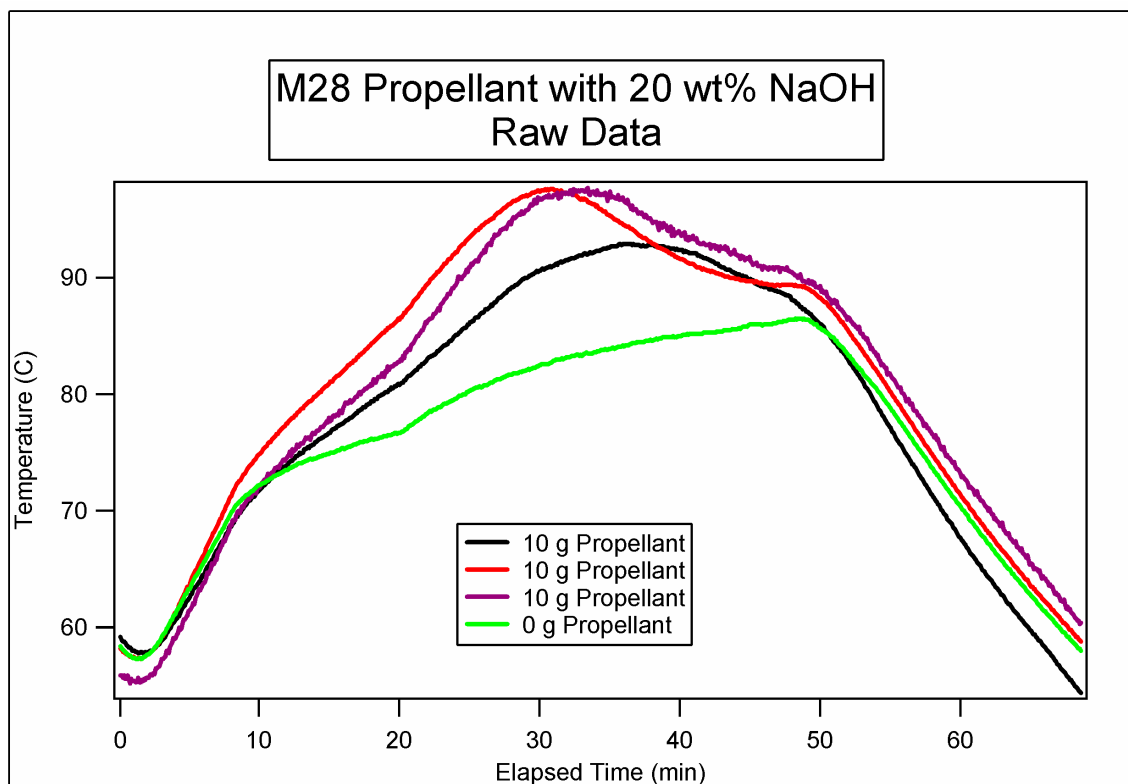


### 3.1.3 M28 Propellant

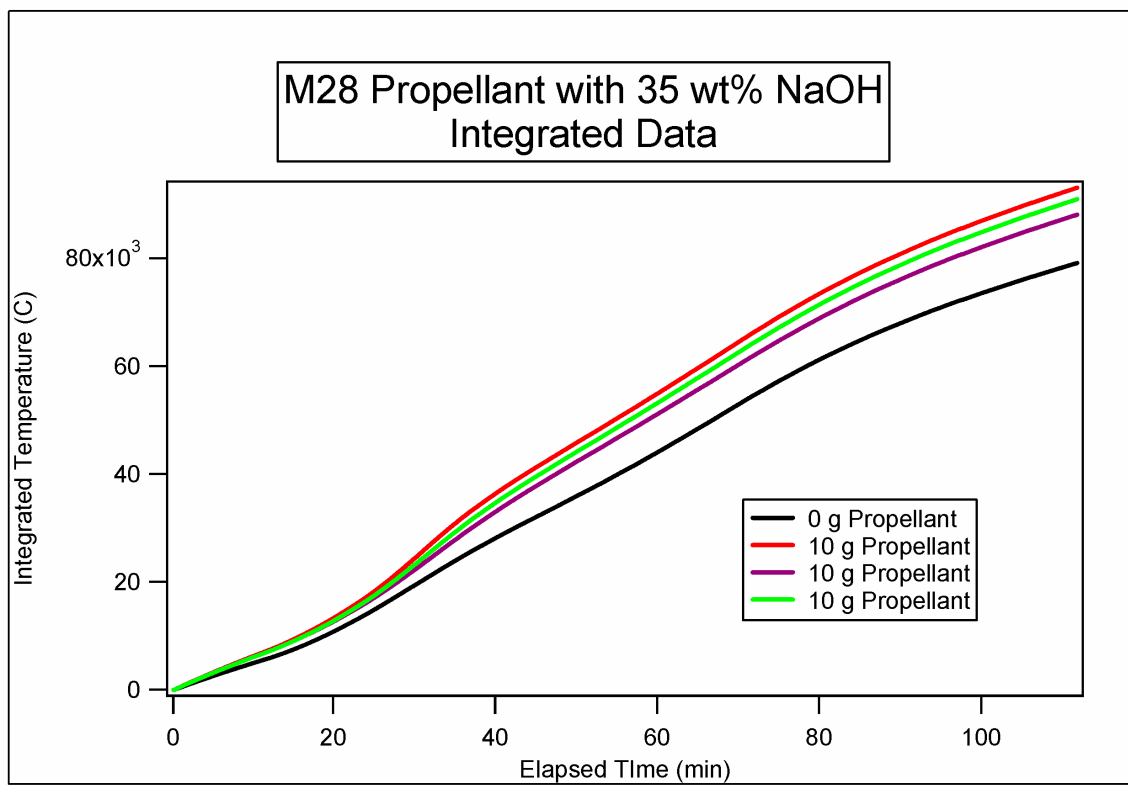
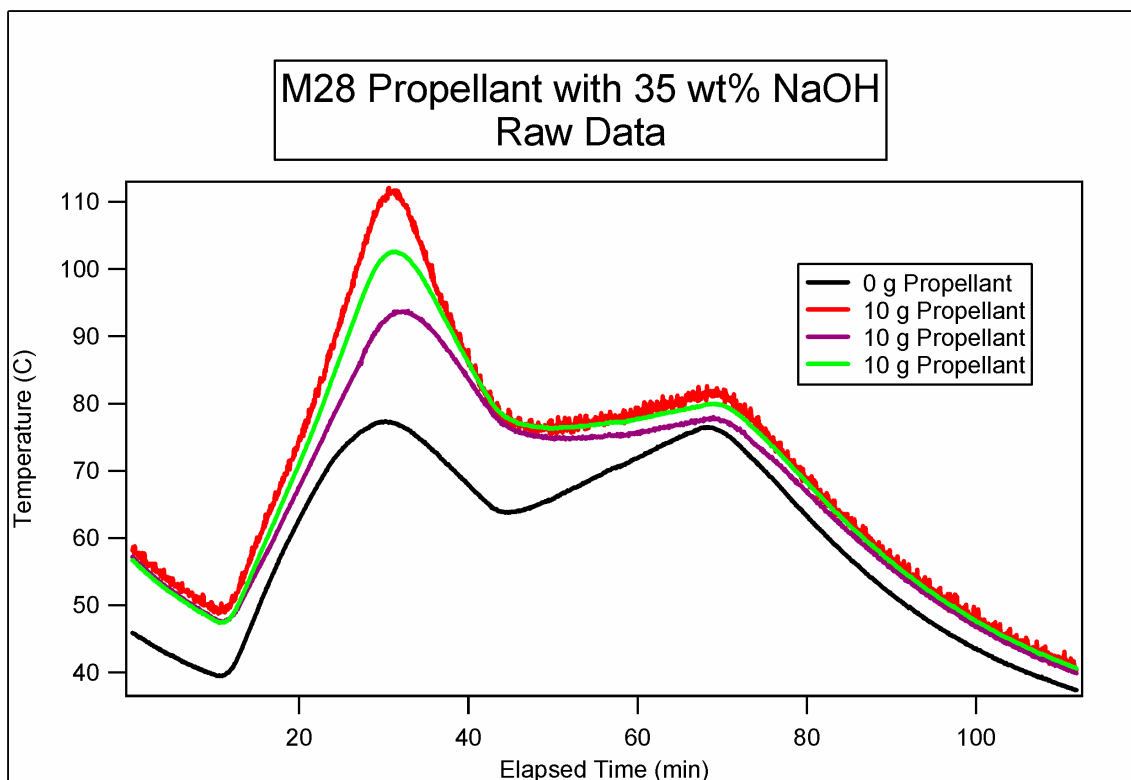
#### 3.1.3.1 12 wt% NaOH, 10 g Propellant/100 g Base



3.1.3.2 20 wt% NaOH, 10 g Propellant/100 g Base

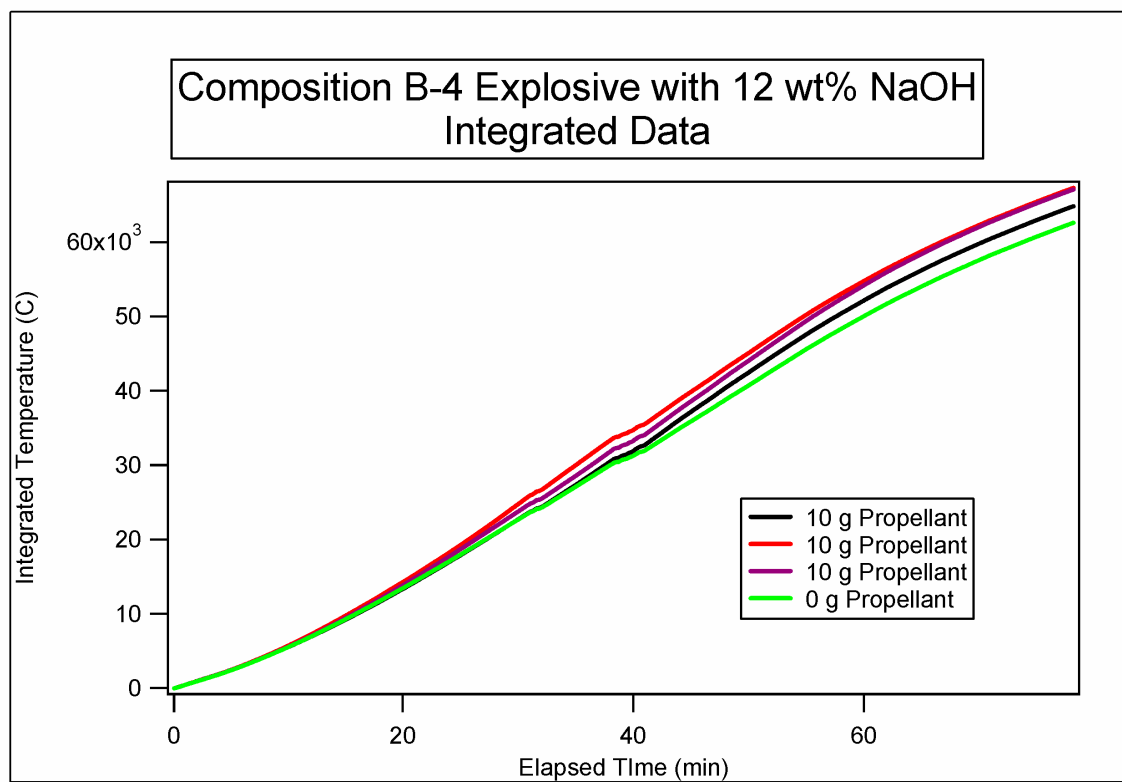
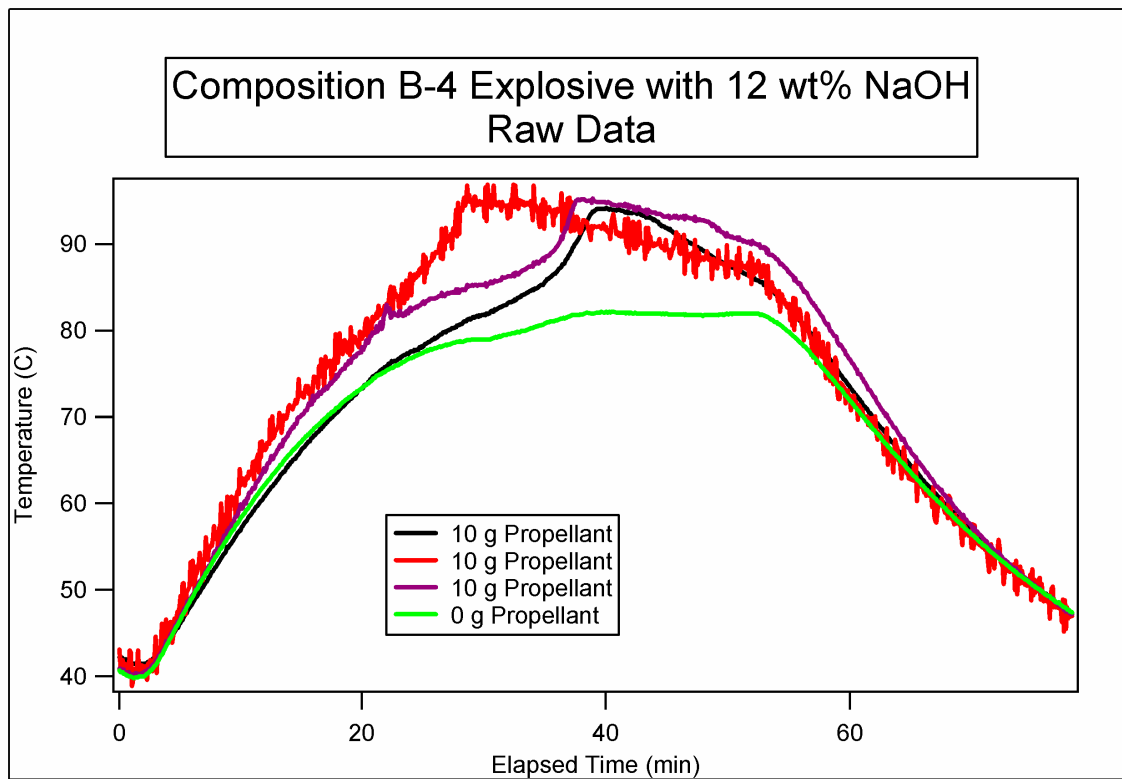


3.1.3.3 35 wt% NaOH, 10 g Propellant/100 g Base

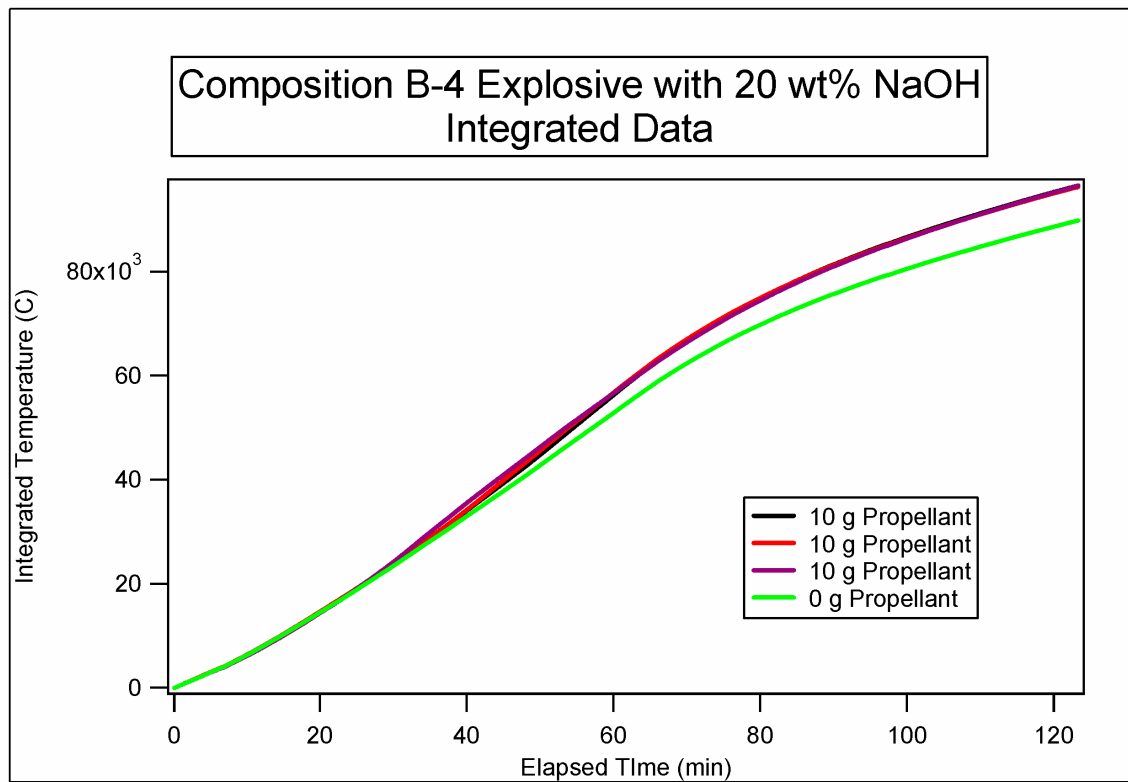
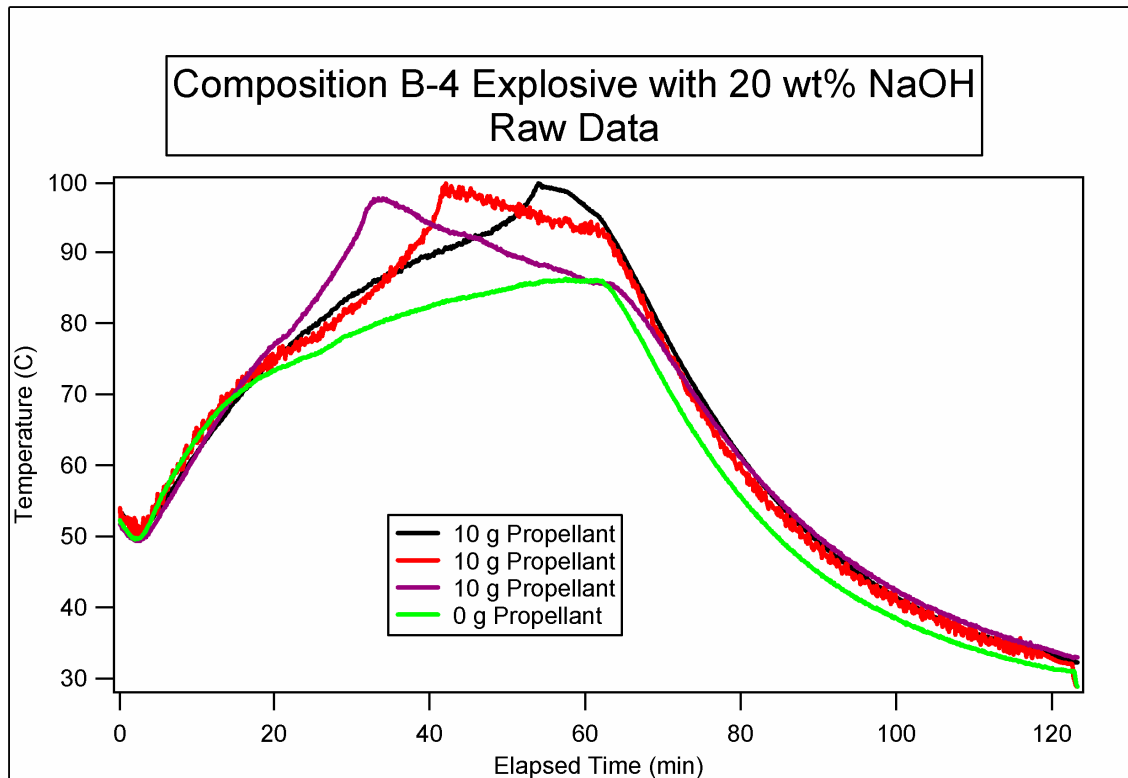


### 3.1.4 Composition B-4

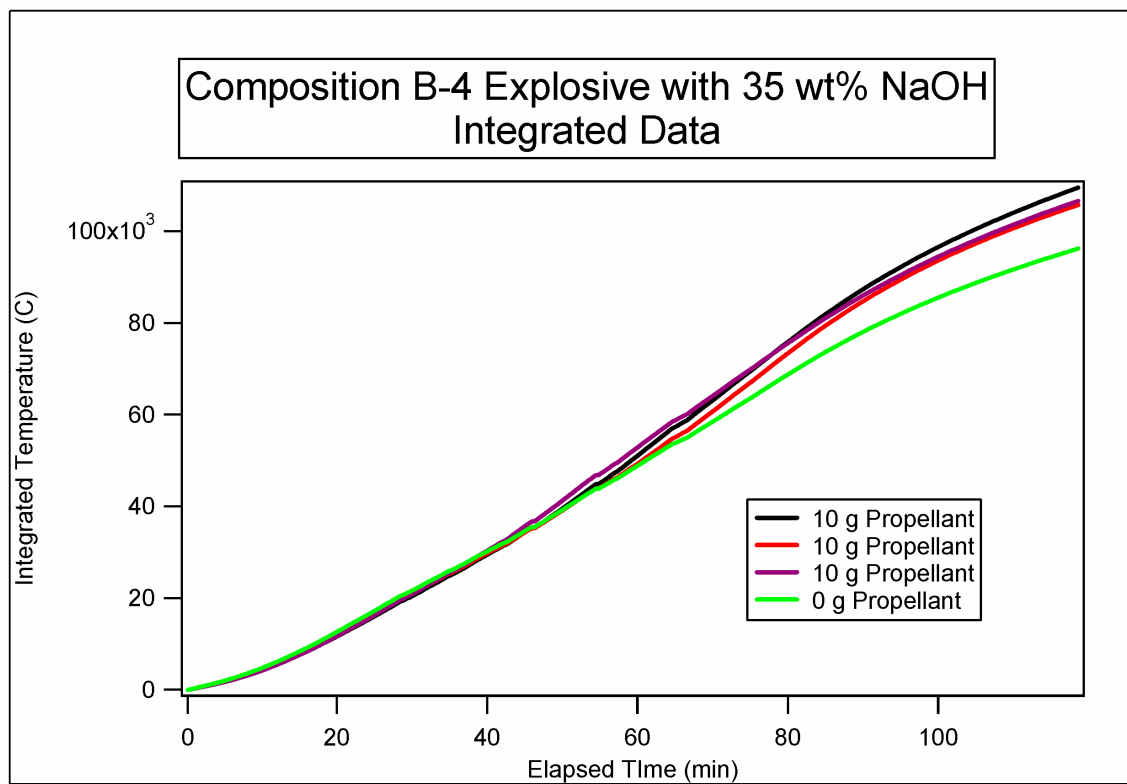
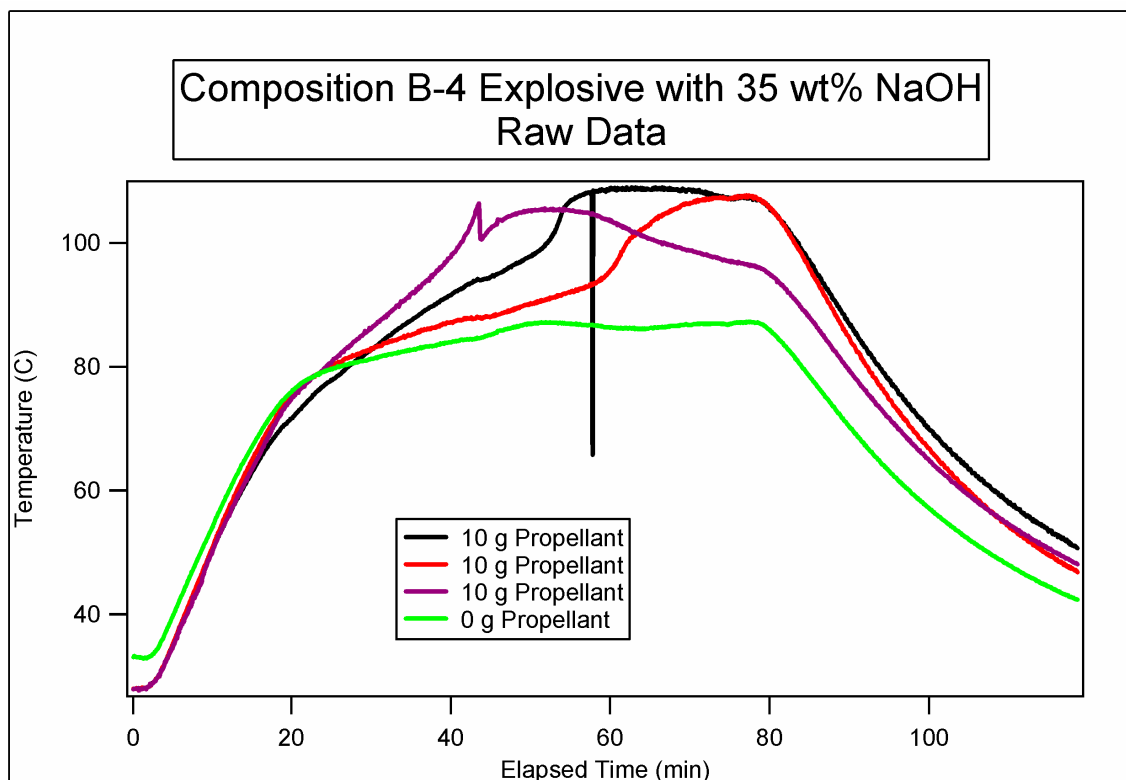
#### 3.1.4.1 12 wt% NaOH, 10 g of Explosive in 100 g Base



3.1.4.2 20 wt% NaOH, 10 g of Explosive in 100 g Base

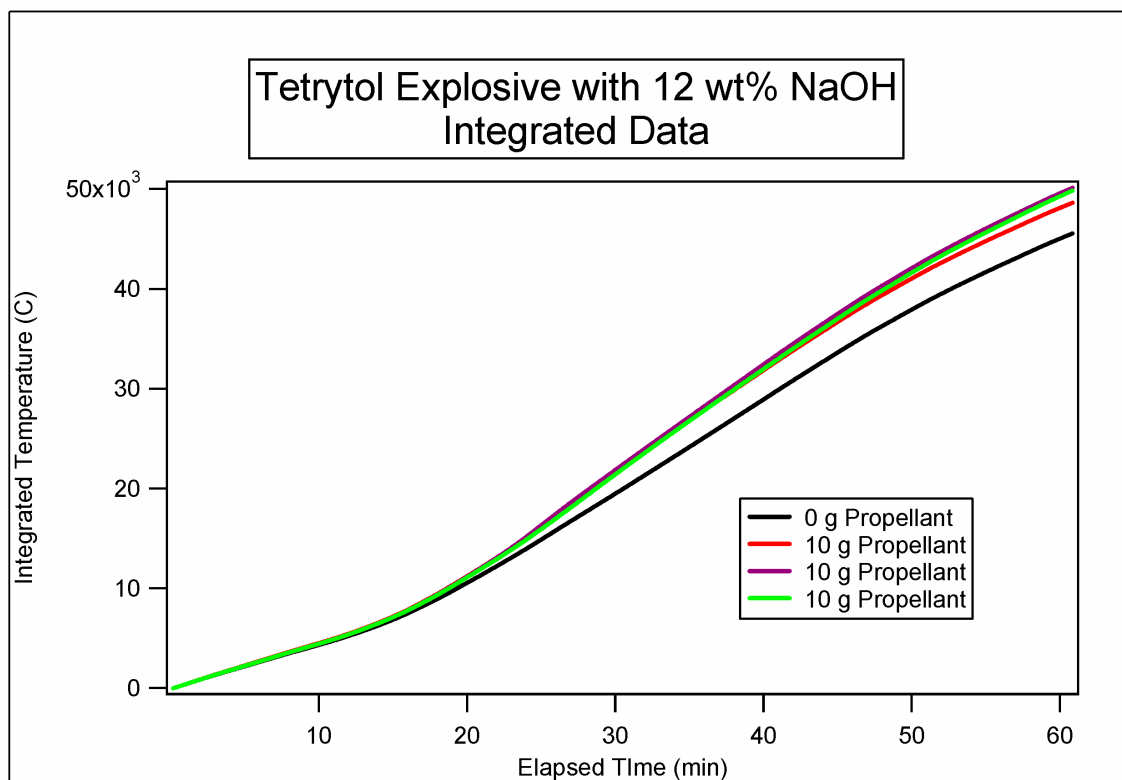
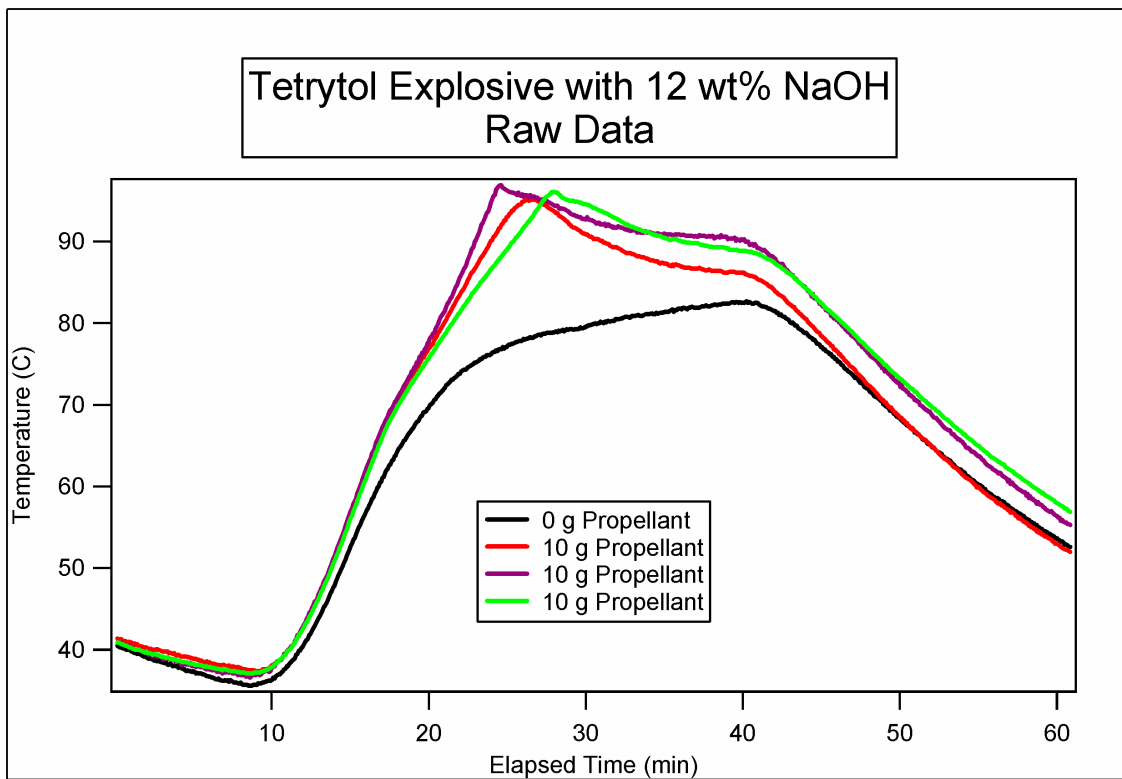


### 3.1.4.3 35 wt% NaOH, 10 g of Explosive in 100 g Base

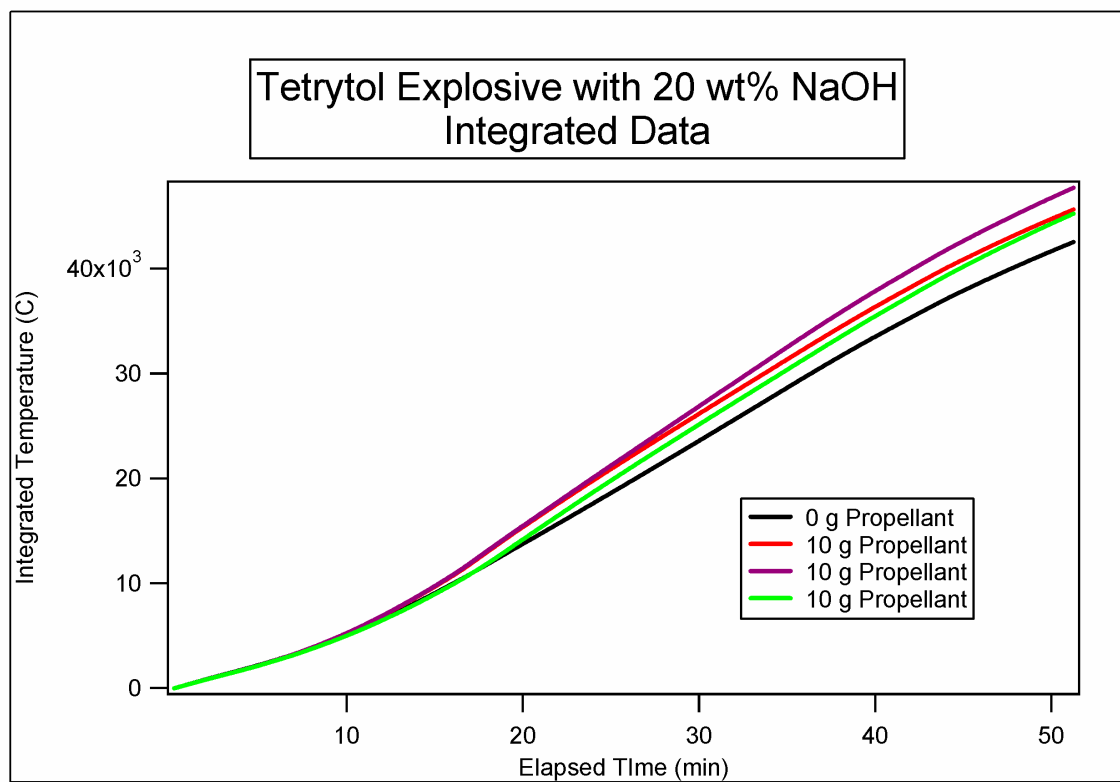
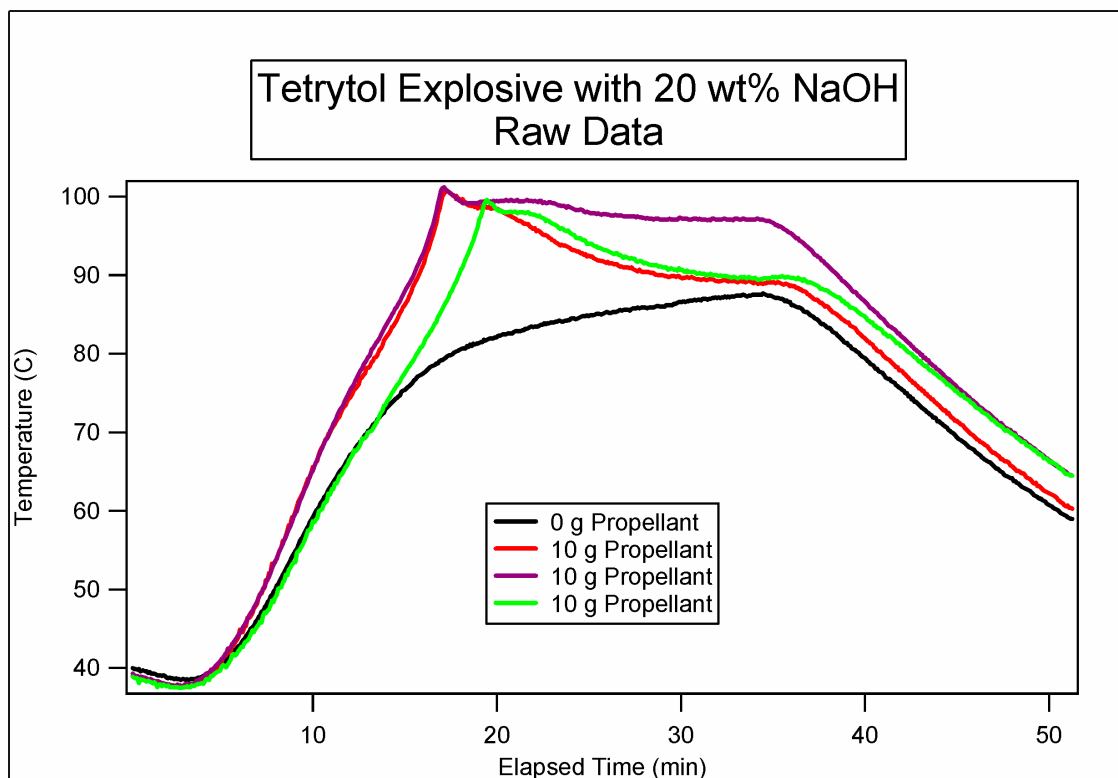


### 3.1.5 Tetrytol

#### 3.1.5.1 12 wt% NaOH, 10 g Explosive in 100 g Base

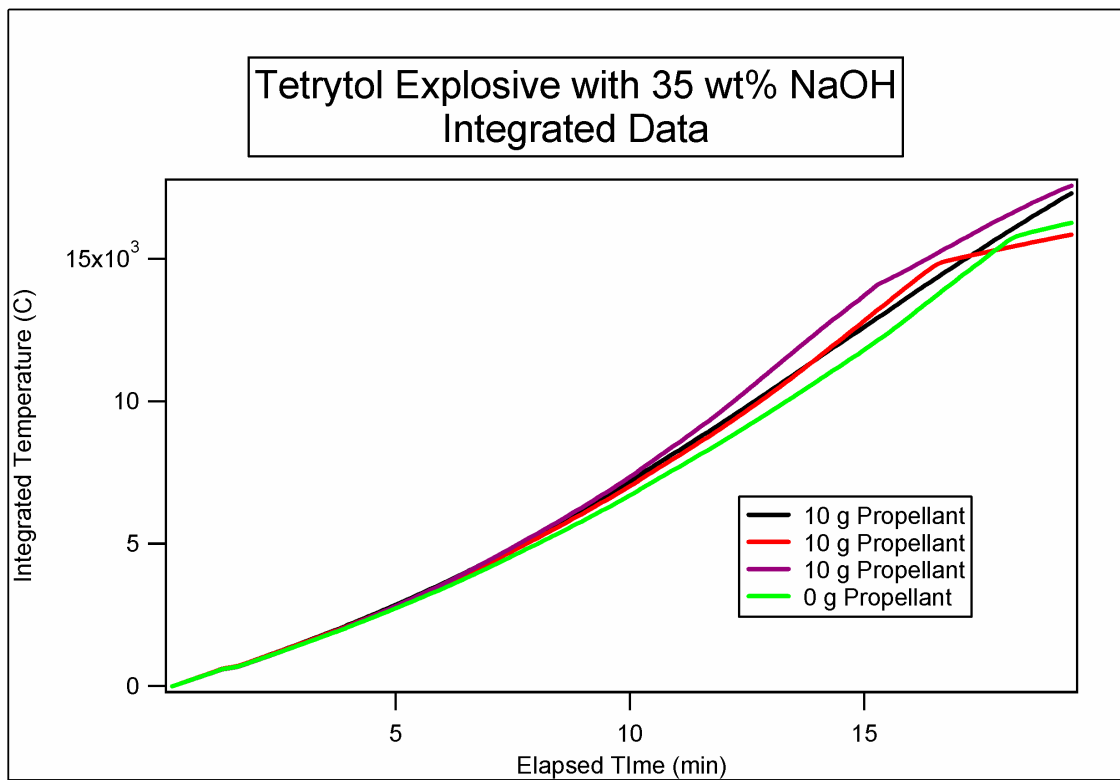
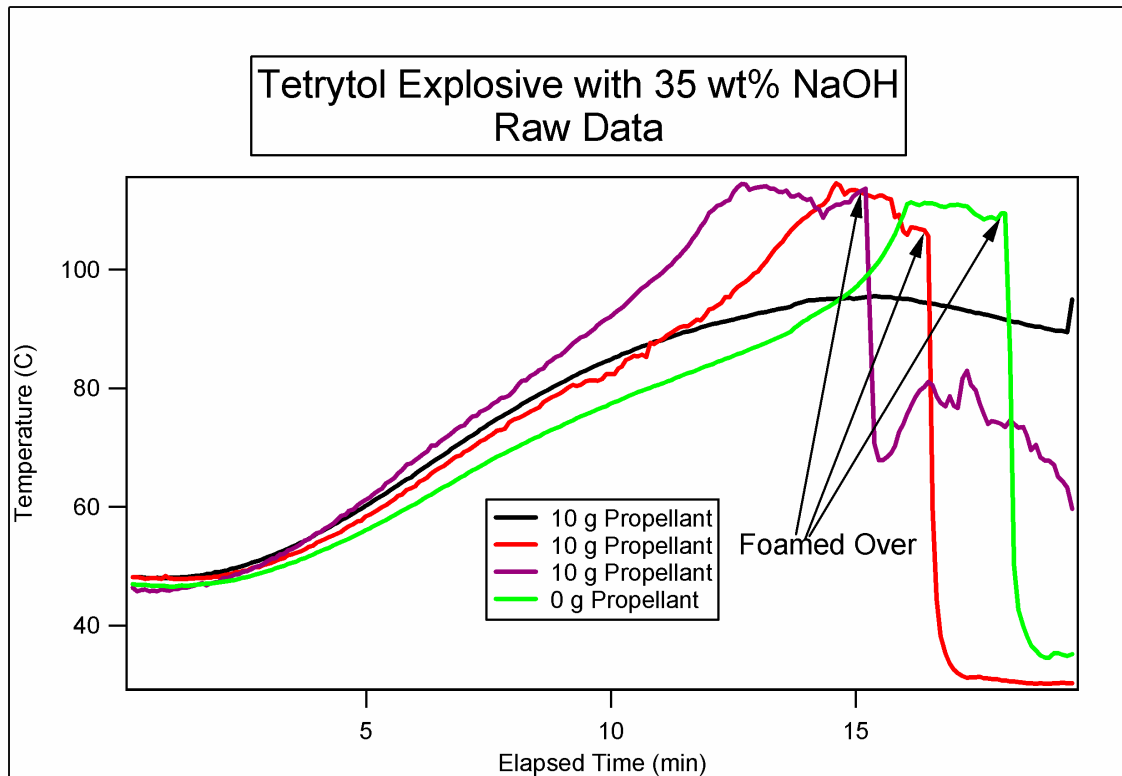


3.1.5.2 20 wt% NaOH, 10 g Explosive in 100 g Base





### 3.1.5.3 20 wt% NaOH, 10 g Explosive in 100 g Base



### 3.2 HEAT OF REACTION SUMMARY TABLE

There are two values listed in the heat of reaction summary table. Heat of reaction averaged over the entire run, and heat of reaction at peak reaction temperature. The second is a higher number and should probably be used for safety and design calculations. For reference, similar studies gave the heat of reaction for HMX at 1.5 kJ/g. Other methods for HMX give values of 2.1 and 2.3 kJ/g. Therefore, this method does not give the most conservative answer. The design number should be estimated as 25-35% higher to account for energy loss due to vaporization and/or boiling.

Explosive	NaOH Concentration	DH <sub>rxn</sub> kJ/g(average) B standard deviation	DH <sub>rxn</sub> kJ/g (peak) B standard deviation
M1	12 wt%	0.151 B 0.009	0.34 B 0.038
M1	20 wt%	0.23 B 0.011	0.59 B 0.018
M1	35 wt%	0.237 B 0.006	1.3 B 0.13
M8	12 wt%	Boiled Over for All Flasks	0.94 B 0.18
M8	20 wt%	0.228 B 0.02	0.89 B 0.09
M8	35 wt%	0.211 B 0.18	1.39 B 0.12
M28	12 wt%	0.115 B 0.005	0.35 B 0.028
M28	20 wt%	0.12 B 0.08	0.38 B 0.14
M28	35 wt%	0.38 B 0.08	1.06 B 0.36
Comp B-4	12 wt%	0.211 B 0.006	0.53 B 0.041
Comp B-4	20 wt%	0.187 B 0.004	0.67 B 0.07
Comp B-4	35 wt%	0.34 B 0.06	0.90 B 0.05
Tetrytol	12 wt%	0.23 B 0.05	0.75 B 0.07
Tetrytol	20 wt%	0.25 B 0.09	0.83 B 0.12
Tetrytol	35 wt%	All Flasks Foamed Over	0.81 B 0.13

### 3.3 OTHER RESULTS

- 1) M1 propellant: Two flasks completely foamed over during the 35 wt% NaOH run. The 12 wt% NaOH was not adequate to complete the reaction of the M1 propellant.
- 2) M8 propellant: The solution boiled over in the 12 wt% NaOH run. For the 20 wt% and 35 wt% experiments, no boiling was observed. A fine tan powder was remaining in the 20 wt% and 35 wt% experiments.
- 3) M28 propellant: Bright orange flakes were apparent on the filter paper for the 20 wt% and the 35 wt% NaOH runs. The 12 wt% NaOH appeared adequate for reaction completion.
- 4) Composition B-4. Some foaming was observed for both the 20 and 35 wt% runs. 12 wt% NaOH was adequate for reaction completion.

- 5) Tetrytol: Every flask foamed over during the 35 wt% NaOH run, making it impossible to get reasonable heat of reaction measurements. The 12 wt% NaOH was adequate for reaction completion.

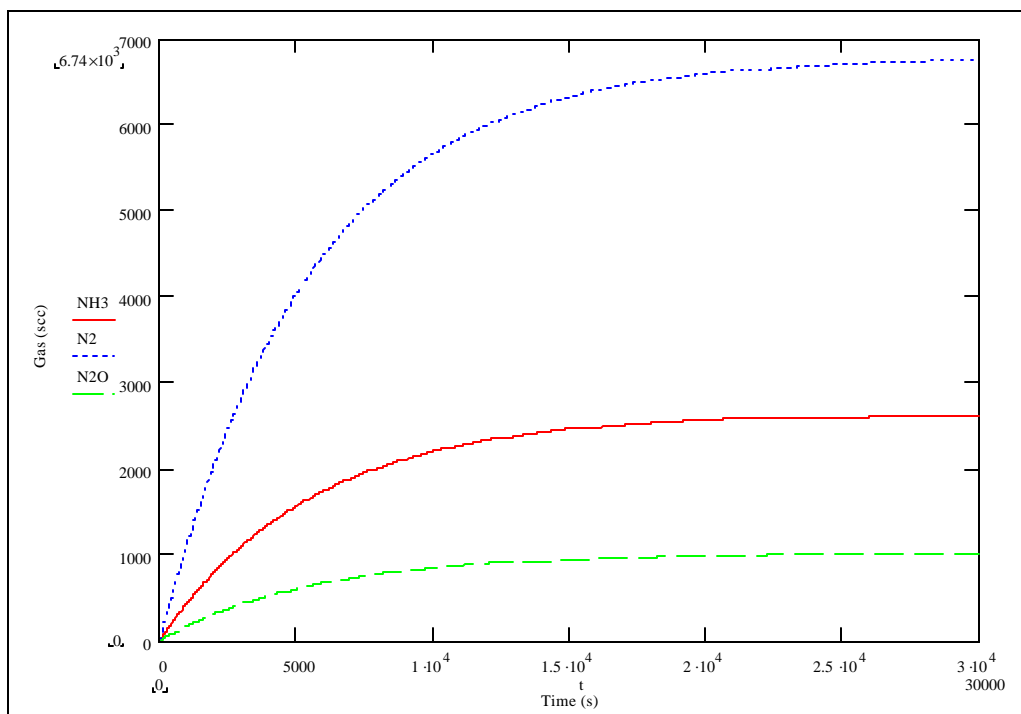
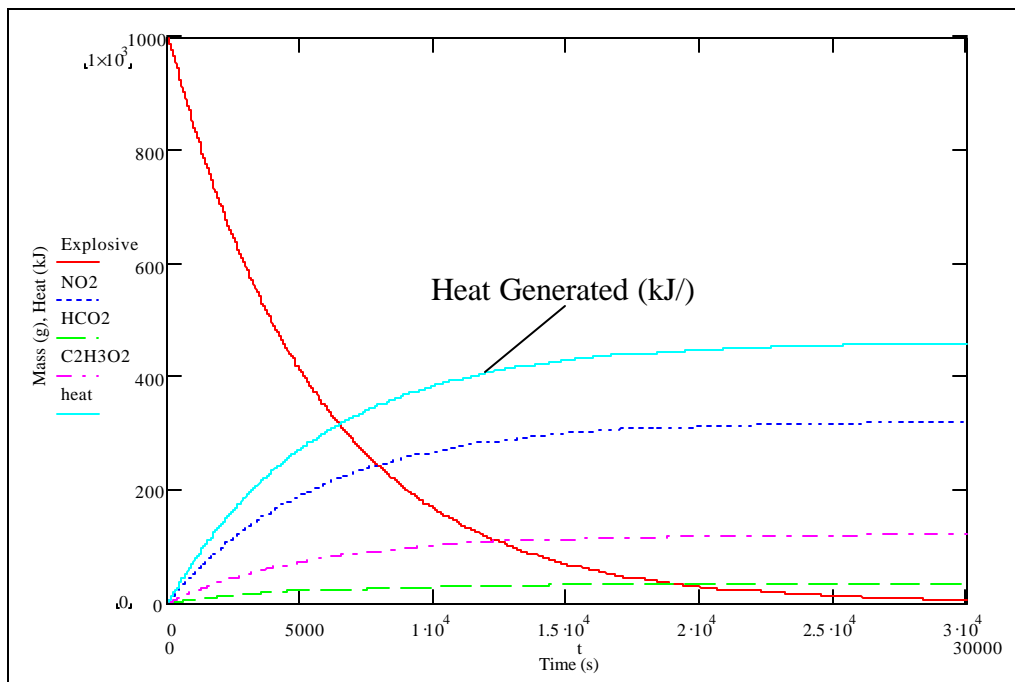
#### **4.0 Reaction Simulations**

Previous data taken from the 2L Parr experiments and the heat of reaction data from these experiments were modeled using a mass-transfer limited kinetic model in Mathcad™. The results of those simulations are shown in the first set of plots. The second set of plots is thermal runaway simulation. Thermal runaway occurs when the heat produced due to reaction is greater than the cooling rate. For the thermal runaway plots, runaway occurs when the heat generation curve is above the cooling lines.

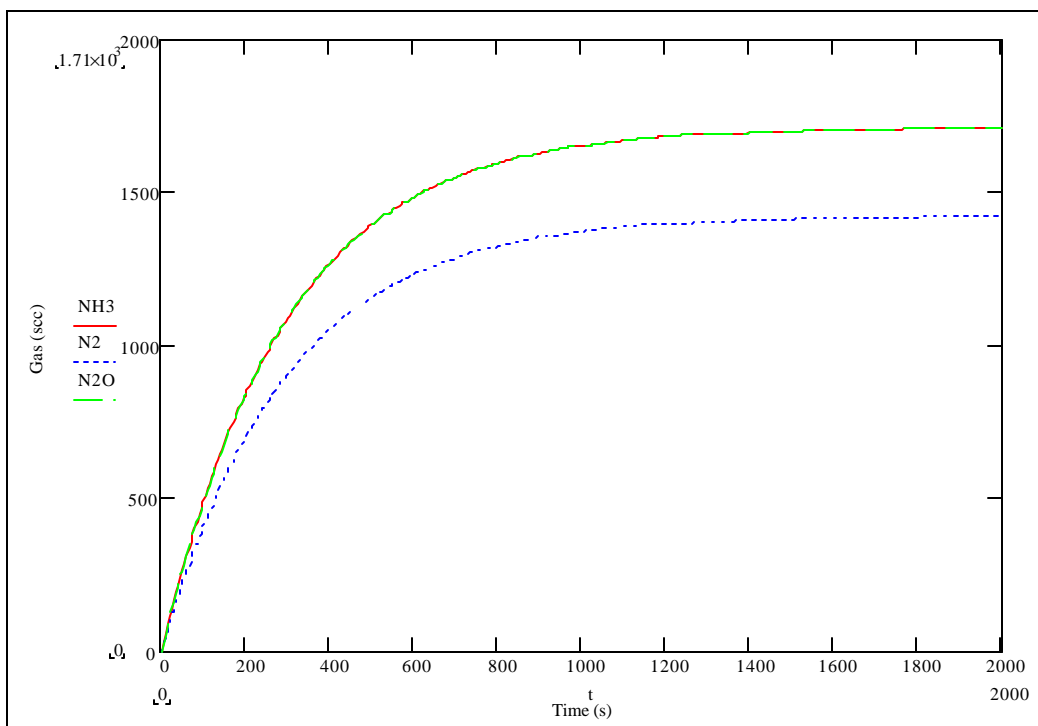
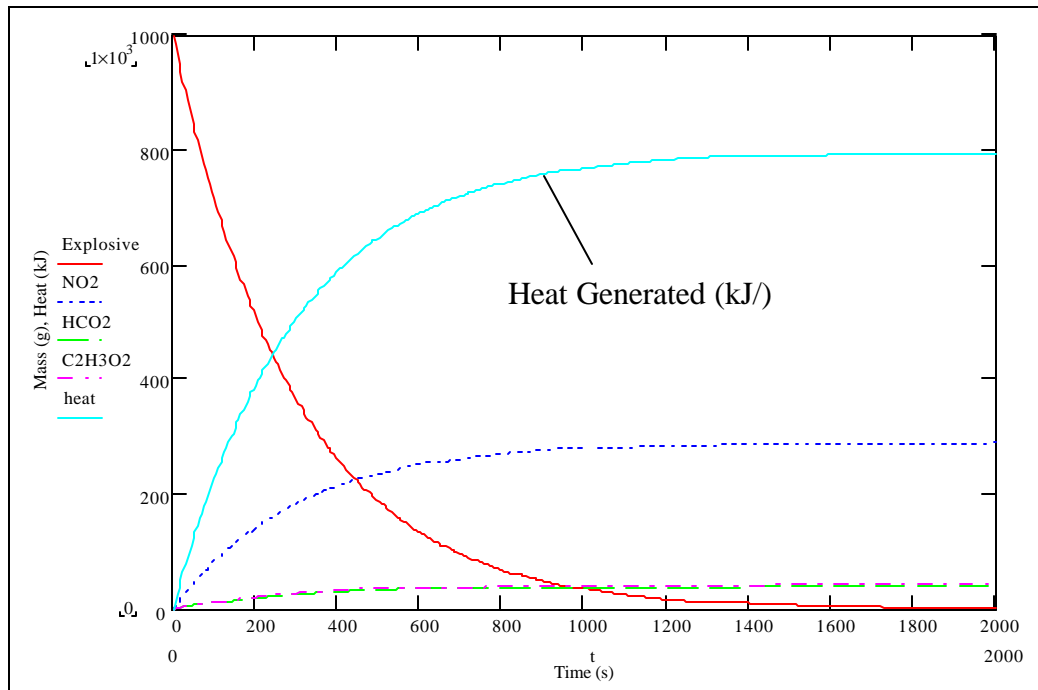
#### 4.1 REACTION PRODUCT AND HEAT MODELING

##### 4.1.1 M1 Propellant

4.1.1.1 12 wt% NaOH, 1000 g Propellant (explosive), 1000 gal of base, Two-84 cm agitator Turning at 100 RPM.

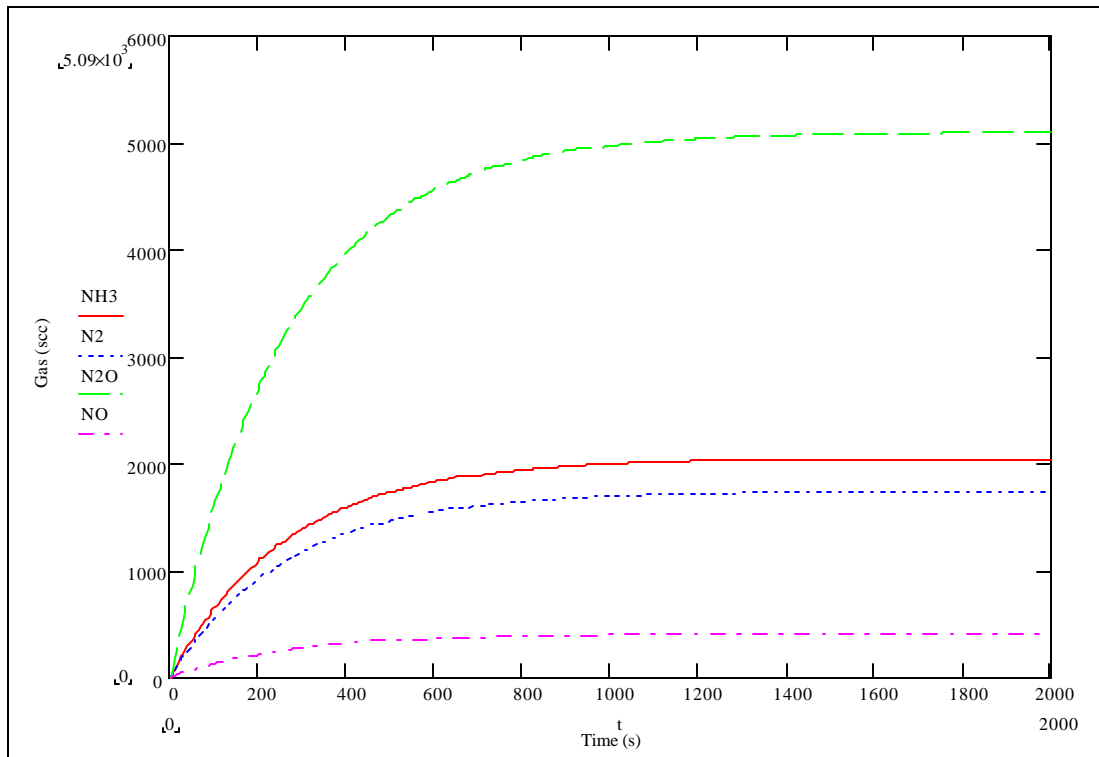
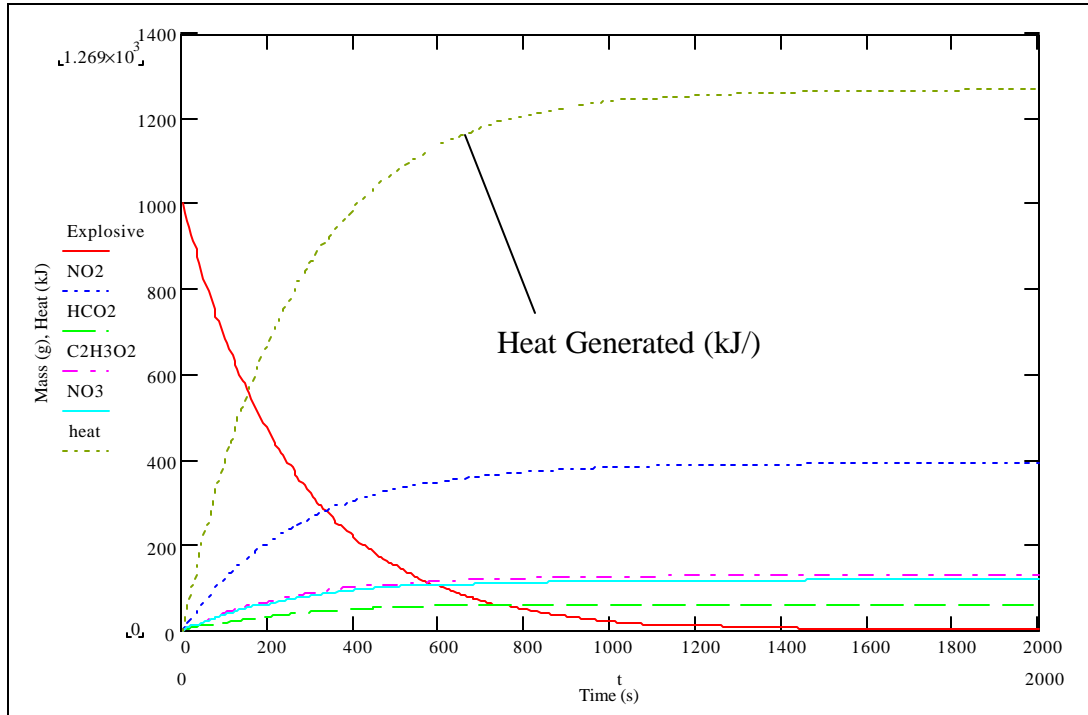


4.1.1.2 20 wt% NaOH, 1000 g Propellant (explosive), 1000 gal of base, Two-84 cm agitator Turning at 100 RPM.

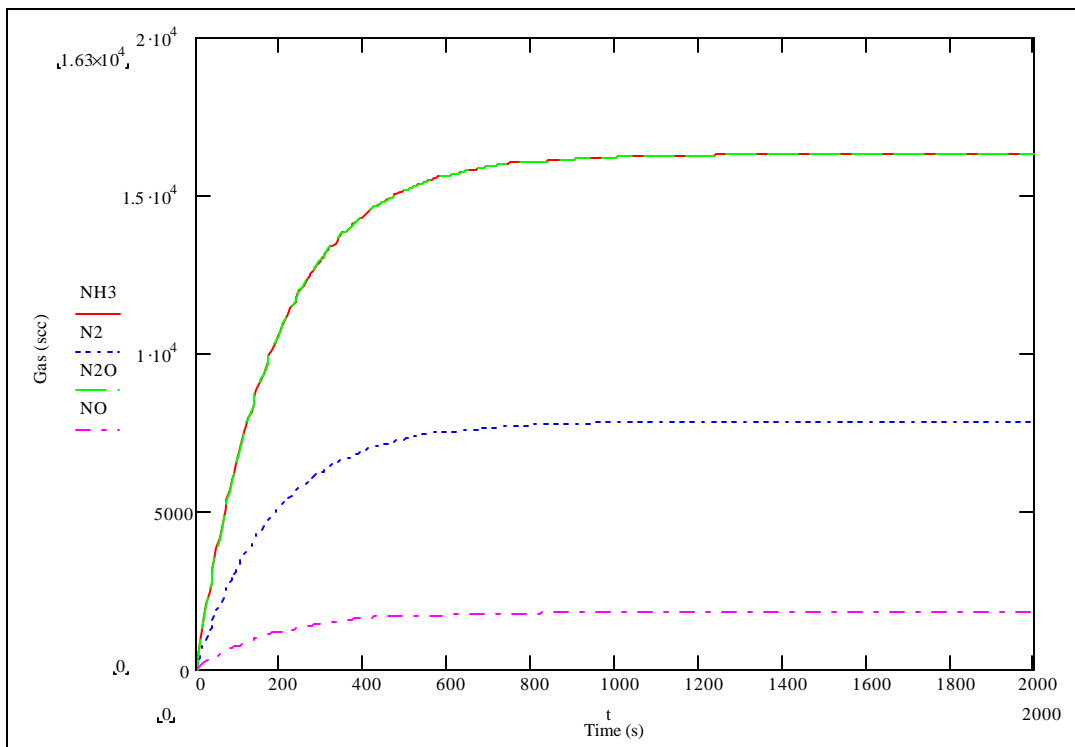
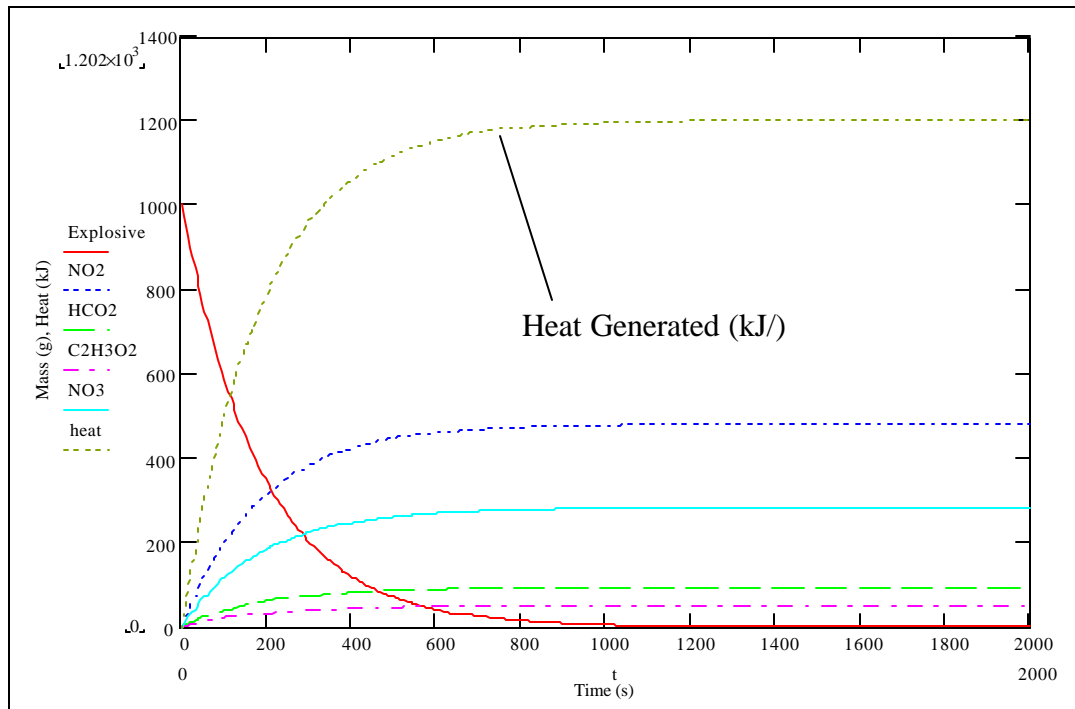


#### 4.1.2 M8 Propellant

4.1.2.1 12 wt% NaOH, 1000 g Propellant (explosive), 1000 gal of base, Two-84 cm agitator Turning at 100 RPM.

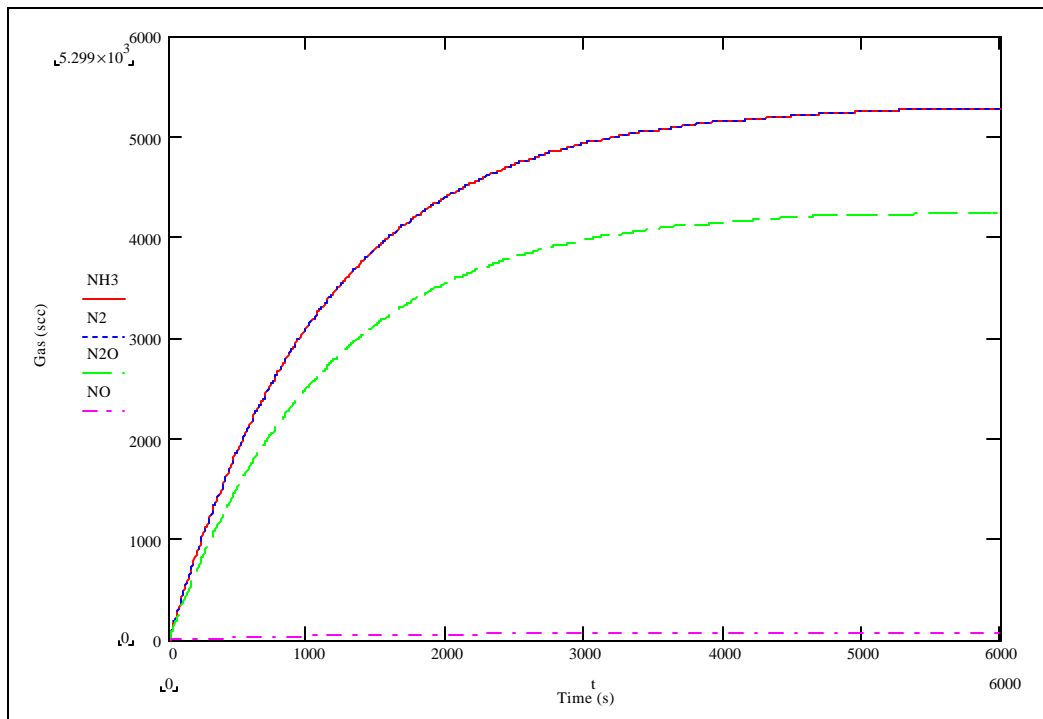
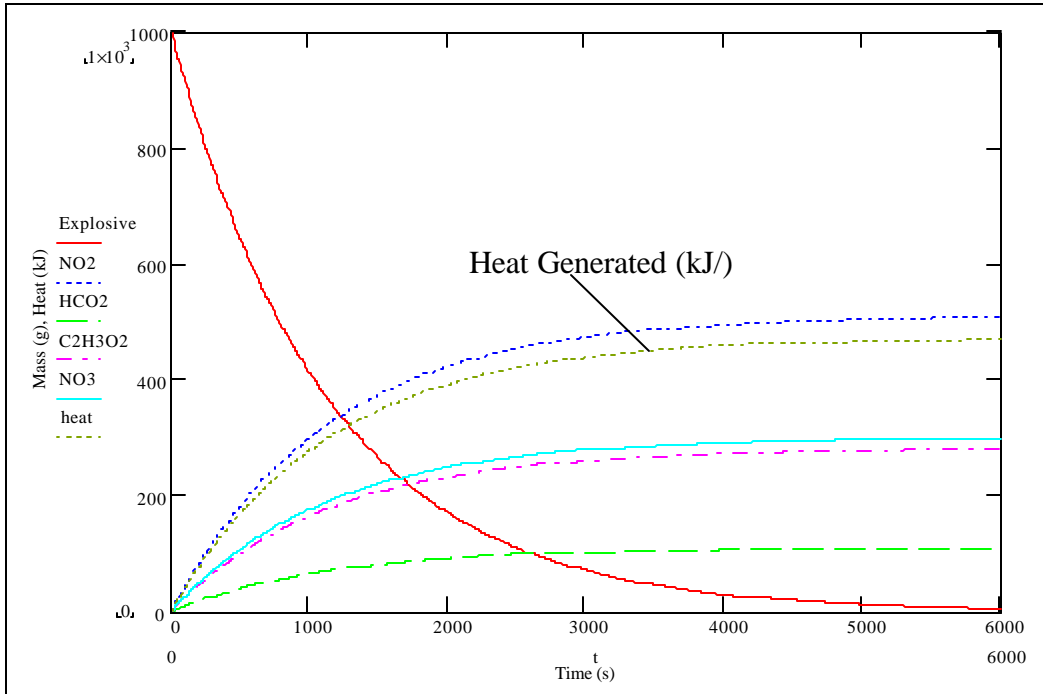


4.1.2.2 20 wt% NaOH, 1000 g Propellant (explosive), 1000 gal of base, Two-84 cm agitator Turning at 100 RPM.



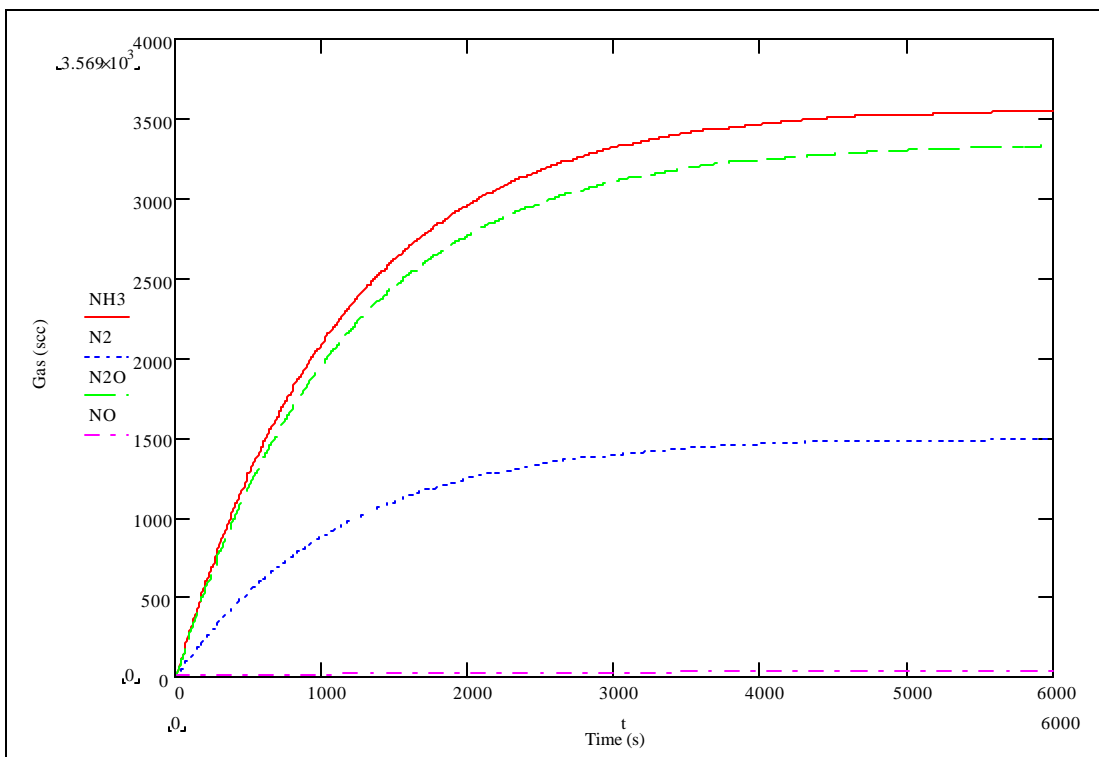
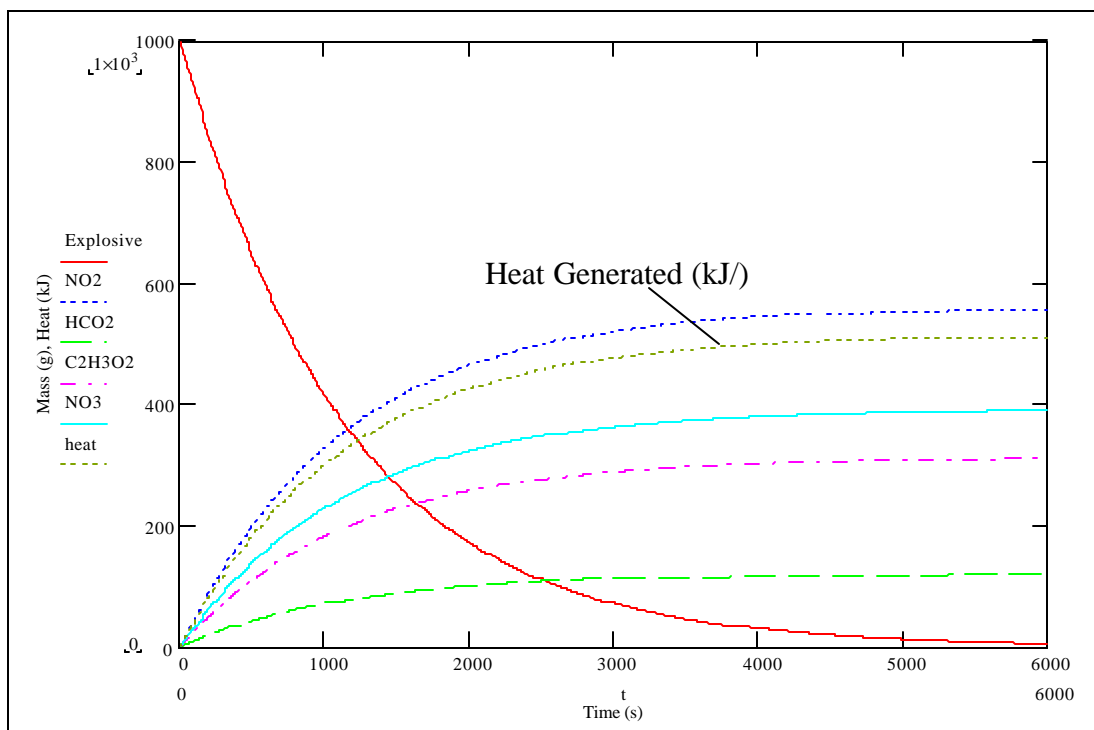
#### 4.1.3 M28 Propellant

4.1.3.1 12 wt% NaOH, 1000 g Propellant (explosive), 1000 gal of base, Two-84 cm agitator Turning at 100 RPM.



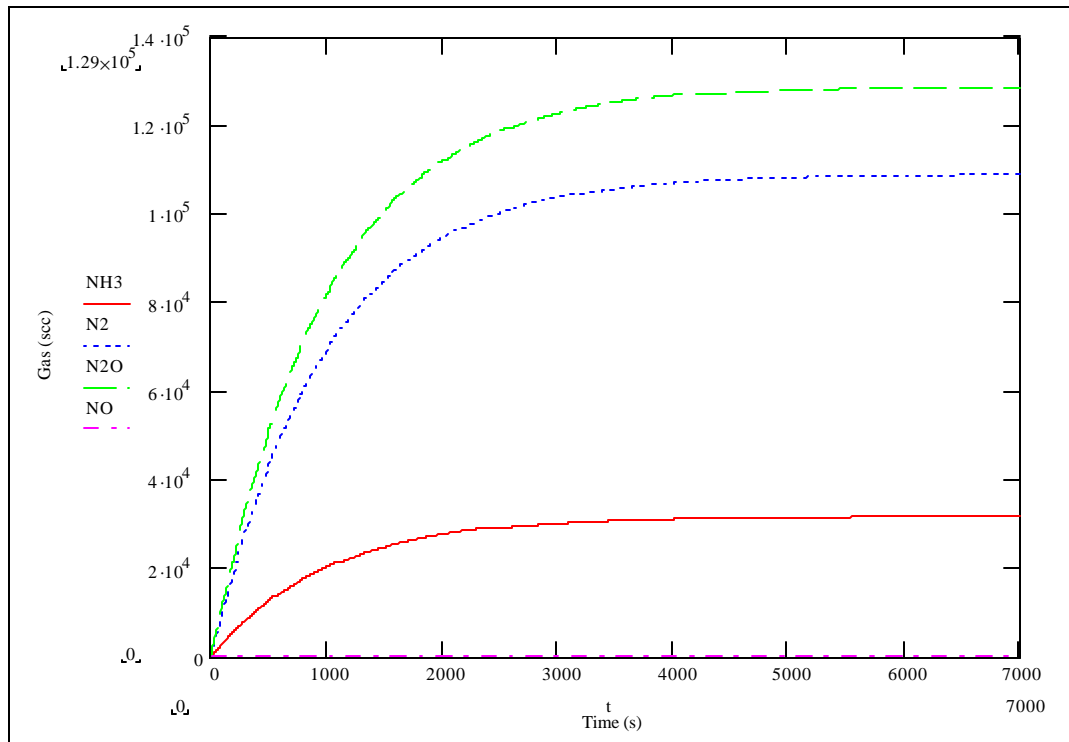
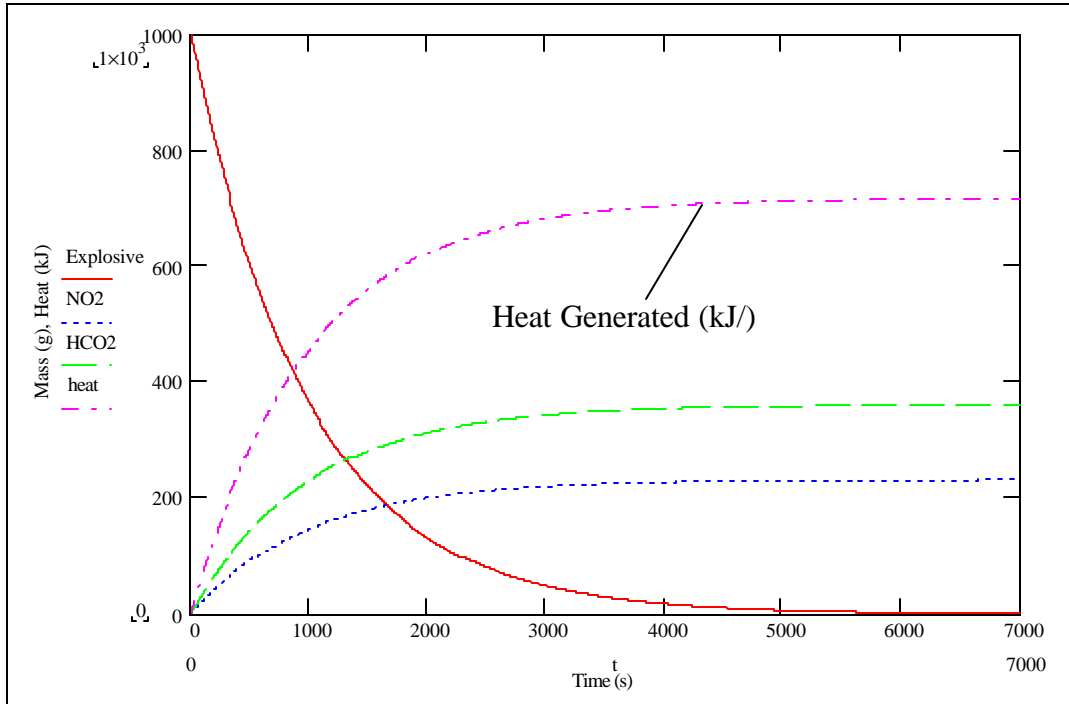


4.1.3.2 20 wt% NaOH, 1000 g Propellant (explosive), 1000 gal of base, Two-84 cm agitator Turning at 100 RPM.

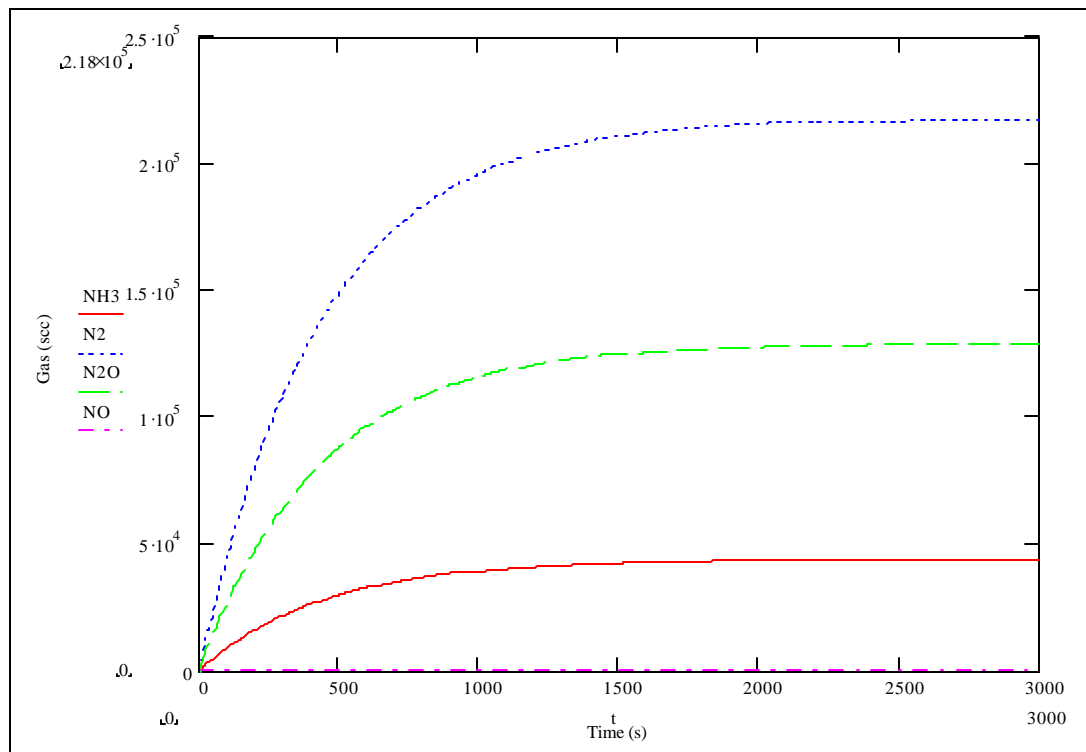
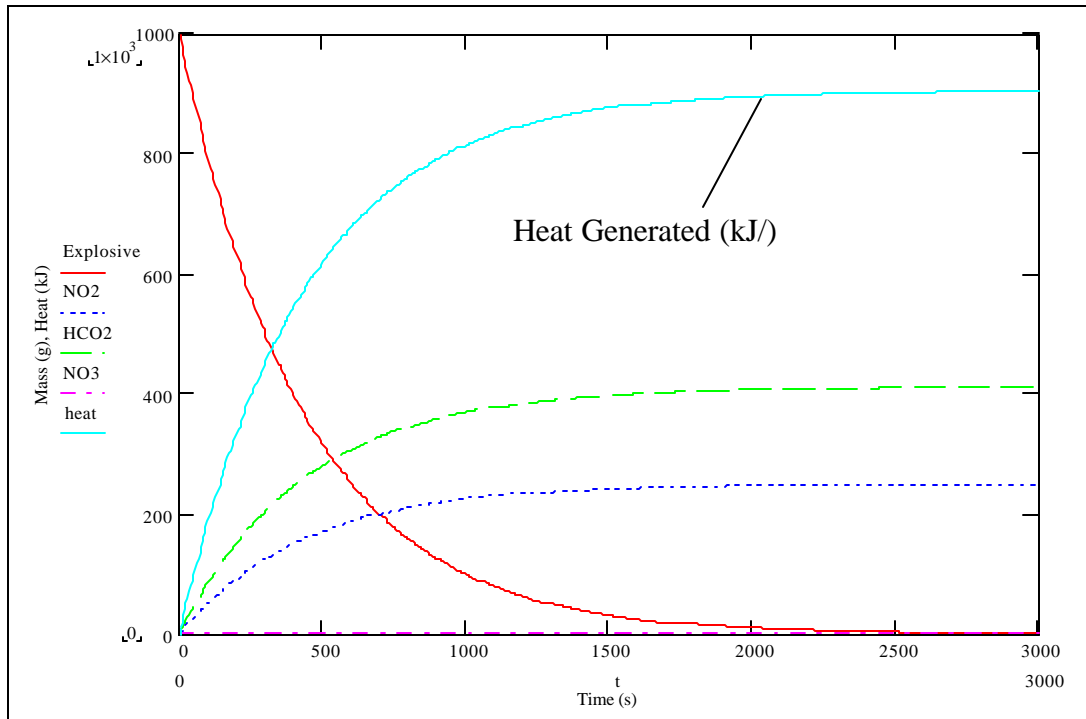


#### 4.1.4 Composition B-4

4.1.4.1 12 wt% NaOH, 1000 g Propellant (explosive), 1000 gal of base, Two-84 cm agitator Turning at 100 RPM.

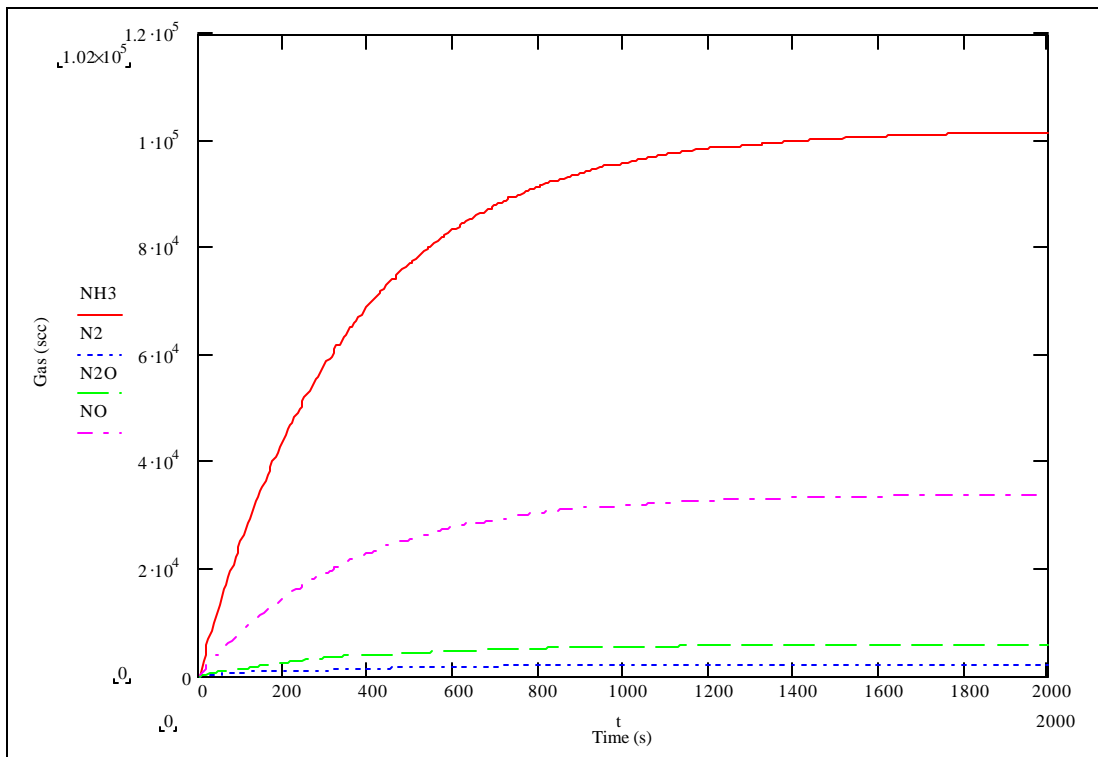
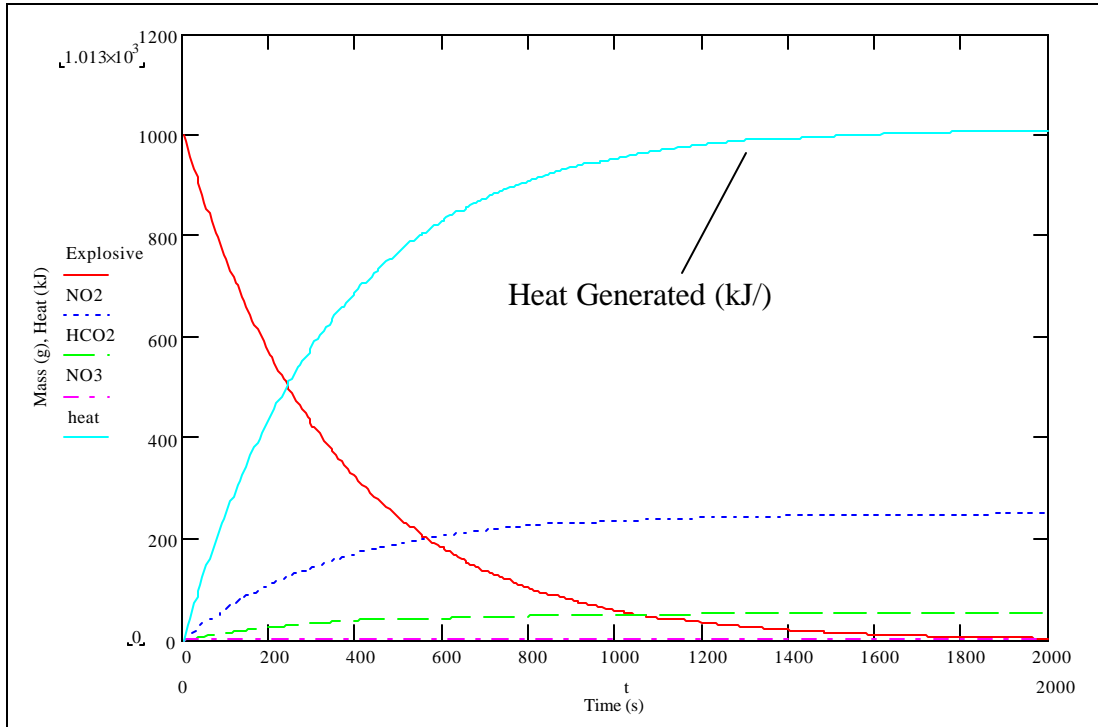


4.1.4.2 20 wt% NaOH, 1000 g Propellant (explosive), 1000 gal of base, Two-84 cm agitator Turning at 100 RPM.

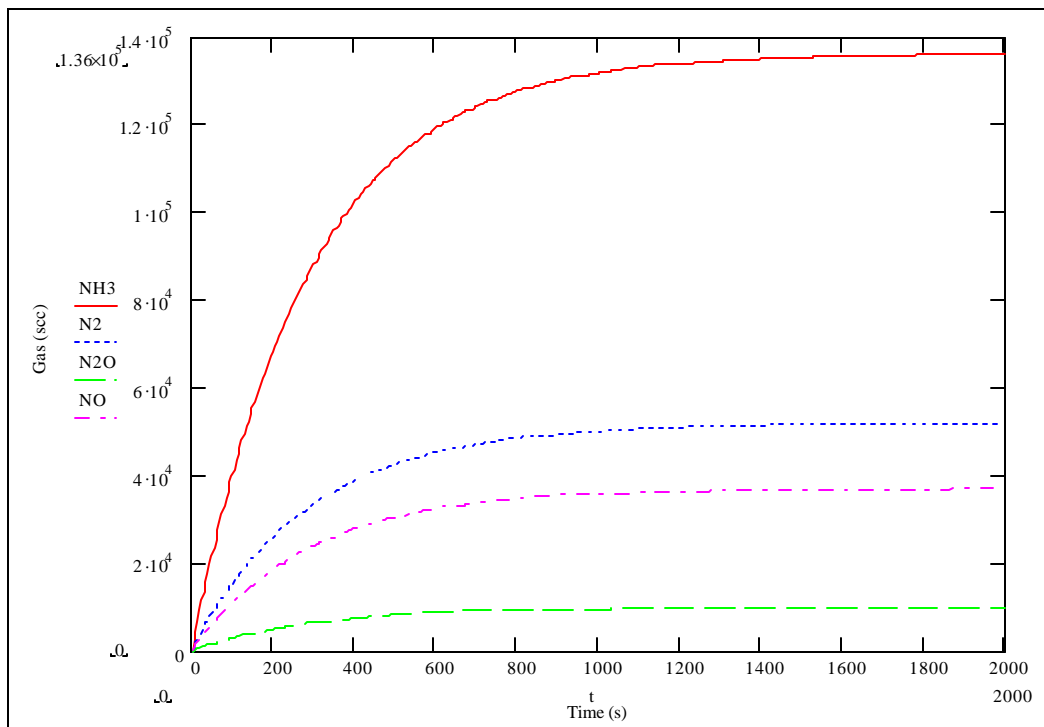
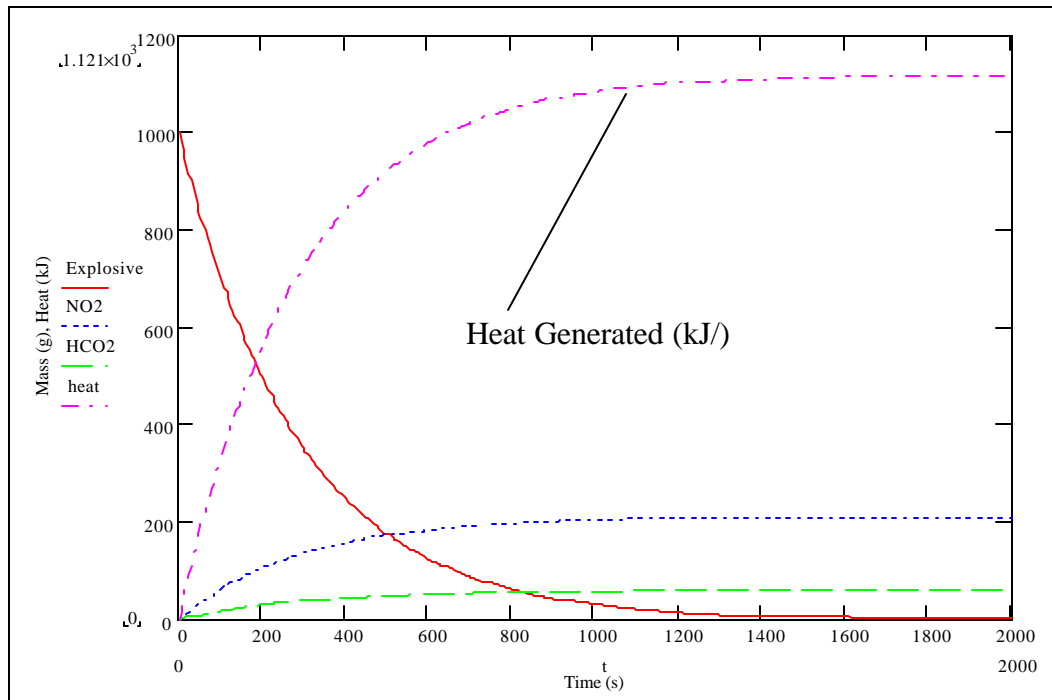


#### 4.1.5 Tetrytol

4.1.5.1 12 wt% NaOH, 1000 g Propellant (explosive), 1000 gal of base, Two-84 cm agitator Turning at 100 RPM.



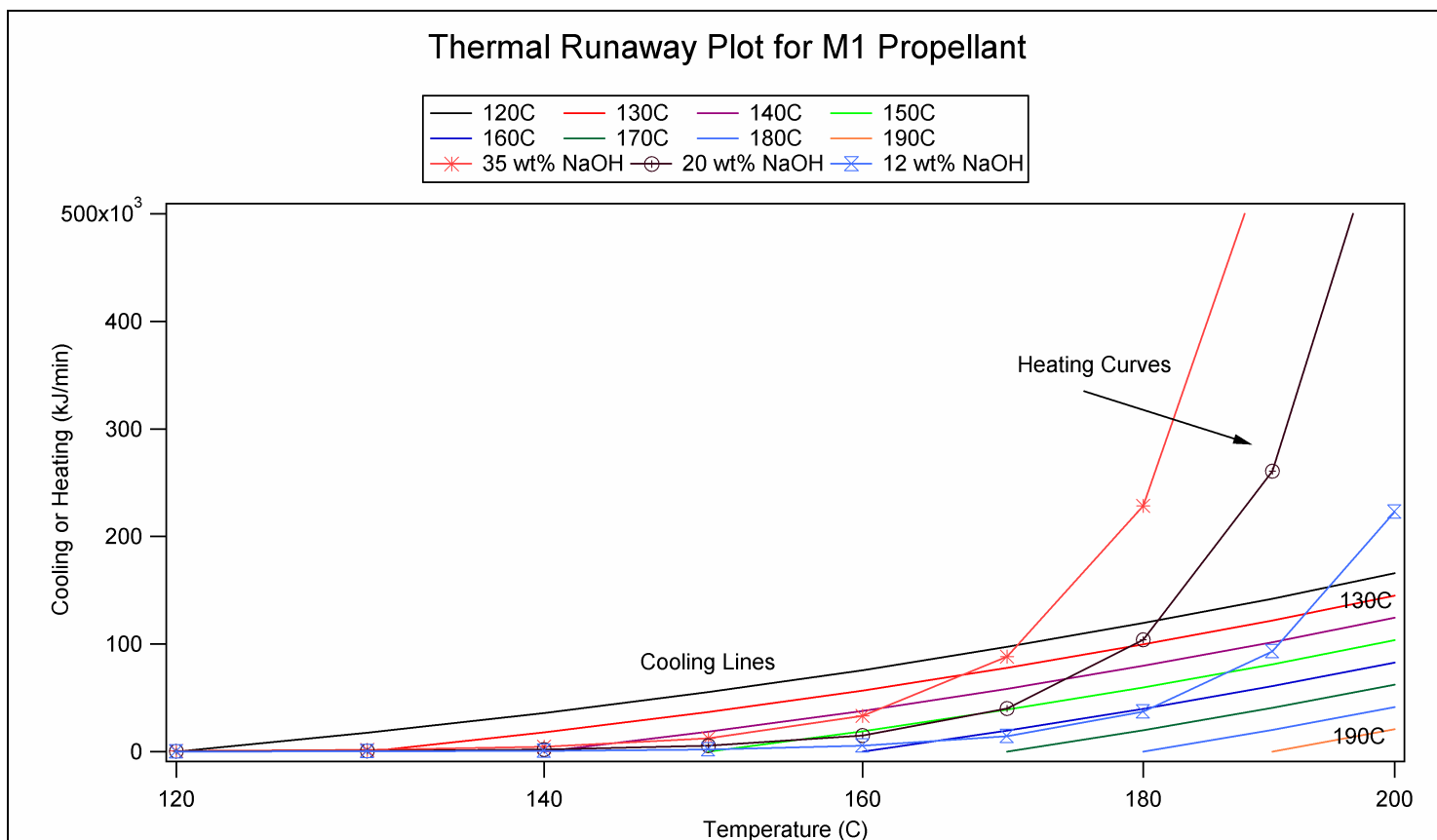
4.1.5.2 20 wt% NaOH, 1000 g Propellant (explosive), 1000 gal of base, Two-84 cm agitator Turning at 100 RPM.



## 4.2 THERMAL RUNAWAY MODELING

### 4.2.1 M1 Propellant

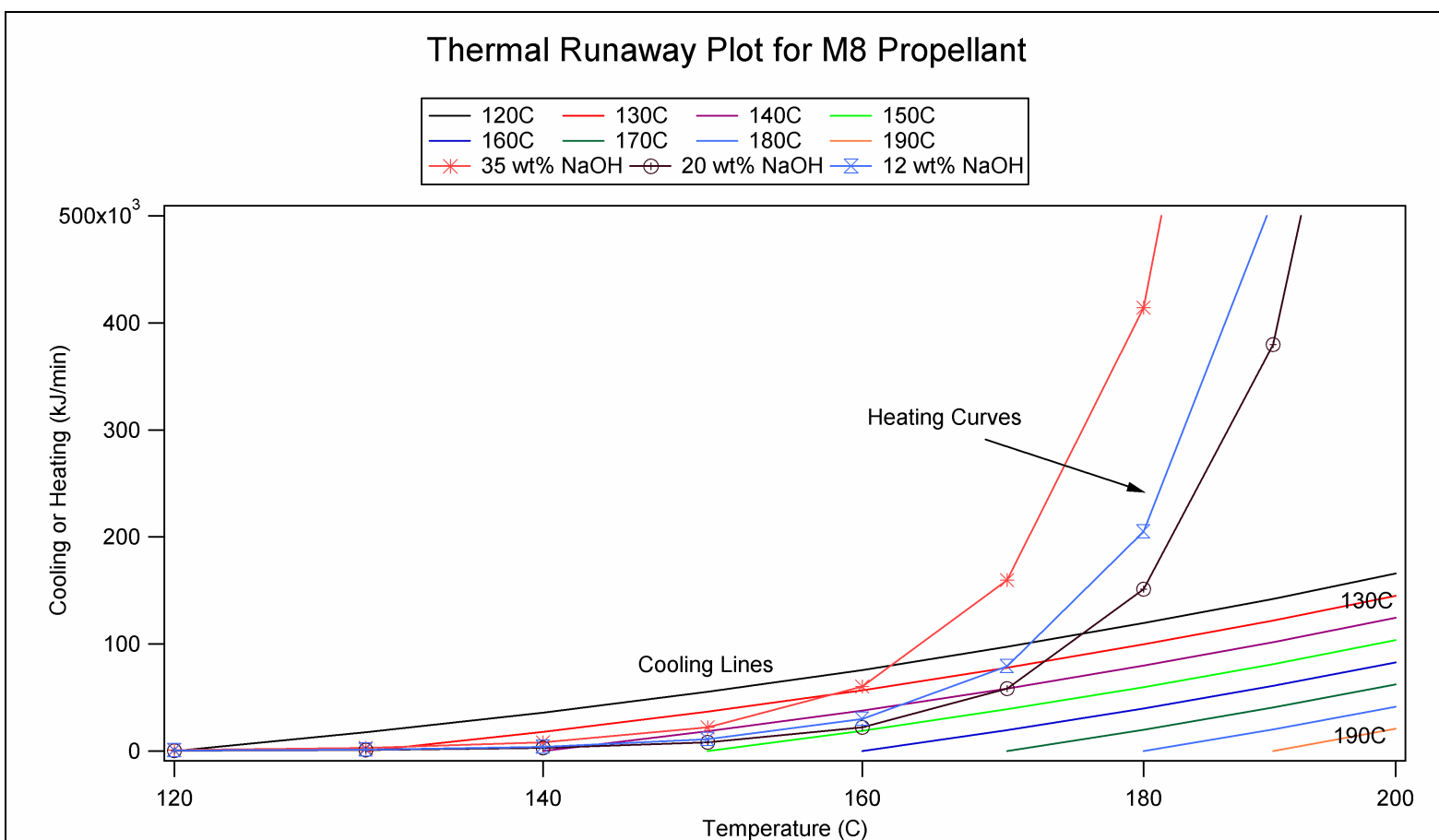
12, 20 and 35 wt% NaOH, 950 g Propellant, 1000 gal of base, Two-84 cm agitator Turning at 800 RPM.



Thermal Runaway at 150°C for 35 wt% NaOH, 160°C for 20 wt% NaOH, and 170°C for 12 wt% NaOH

#### 4.2.2 M8 Propellant

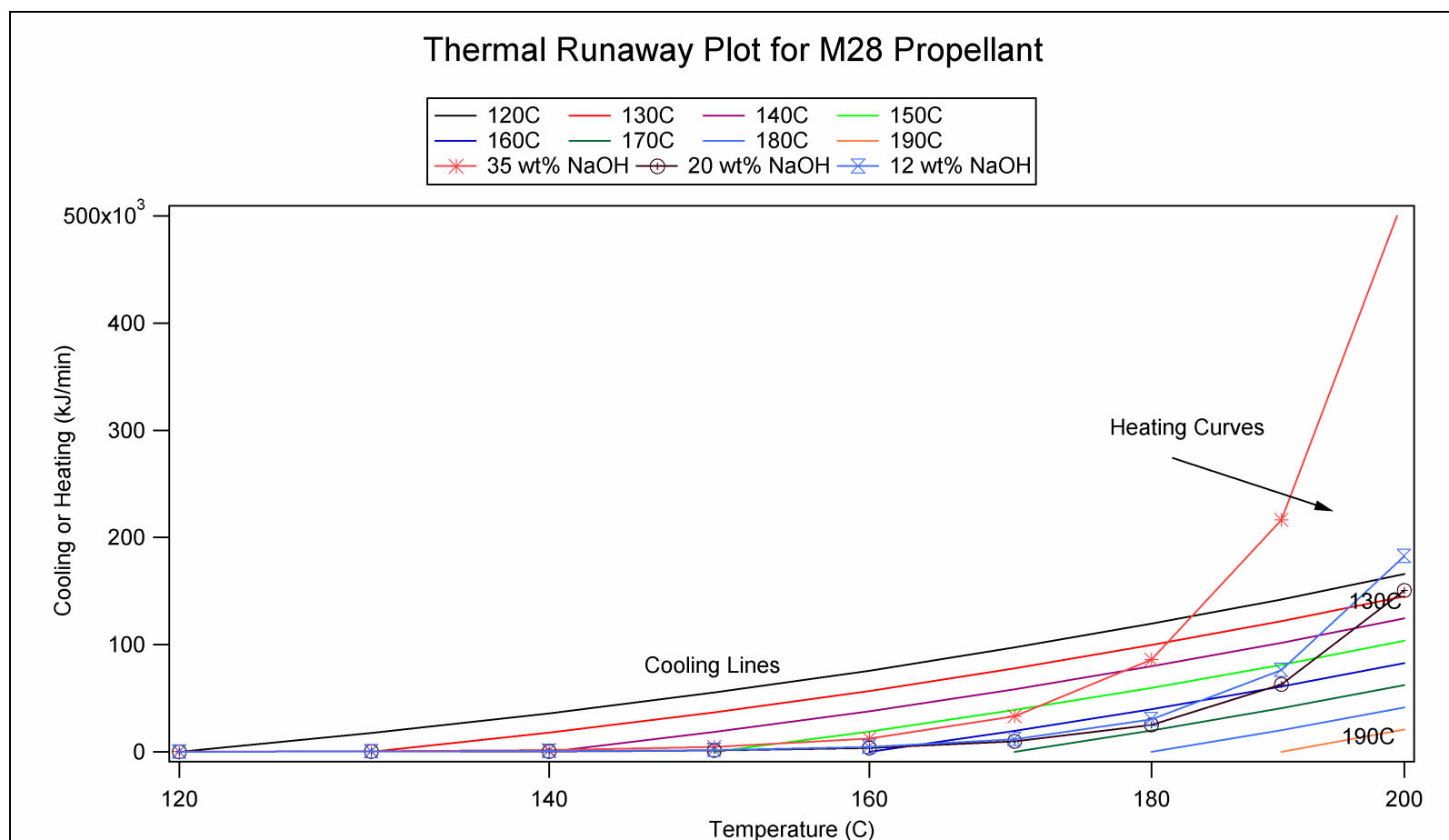
12, 20 and 35 wt% NaOH, 950 g Propellant, 1000 gal of base, Two-84 cm agitator Turning at 800 RPM.



Thermal Runaway at 130°C for 35 wt% NaOH, 140°C for 20 wt% NaOH, and 150°C for 12 wt% NaOH

#### 4.2.3 M28 Propellant

12, 20 and 35 wt% NaOH, 950 g Propellant, 1000 gal of base, Two-84 cm agitator Turning at 800 RPM.

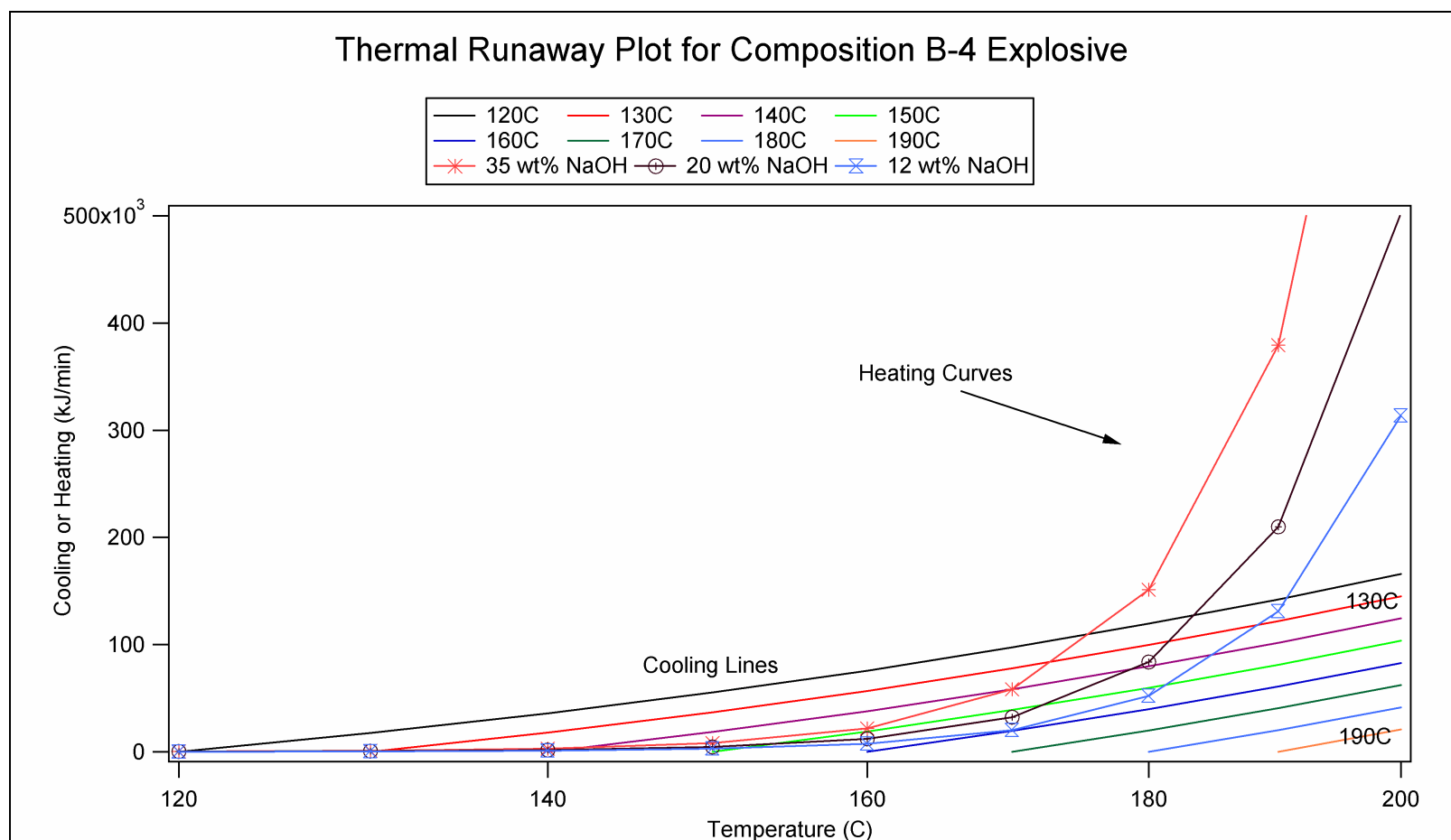


Thermal Runaway at 160°C for 35 wt% NaOH, 170°C for 20 wt% NaOH and 12 wt% NaOH



#### 4.2.4 Composition B-4

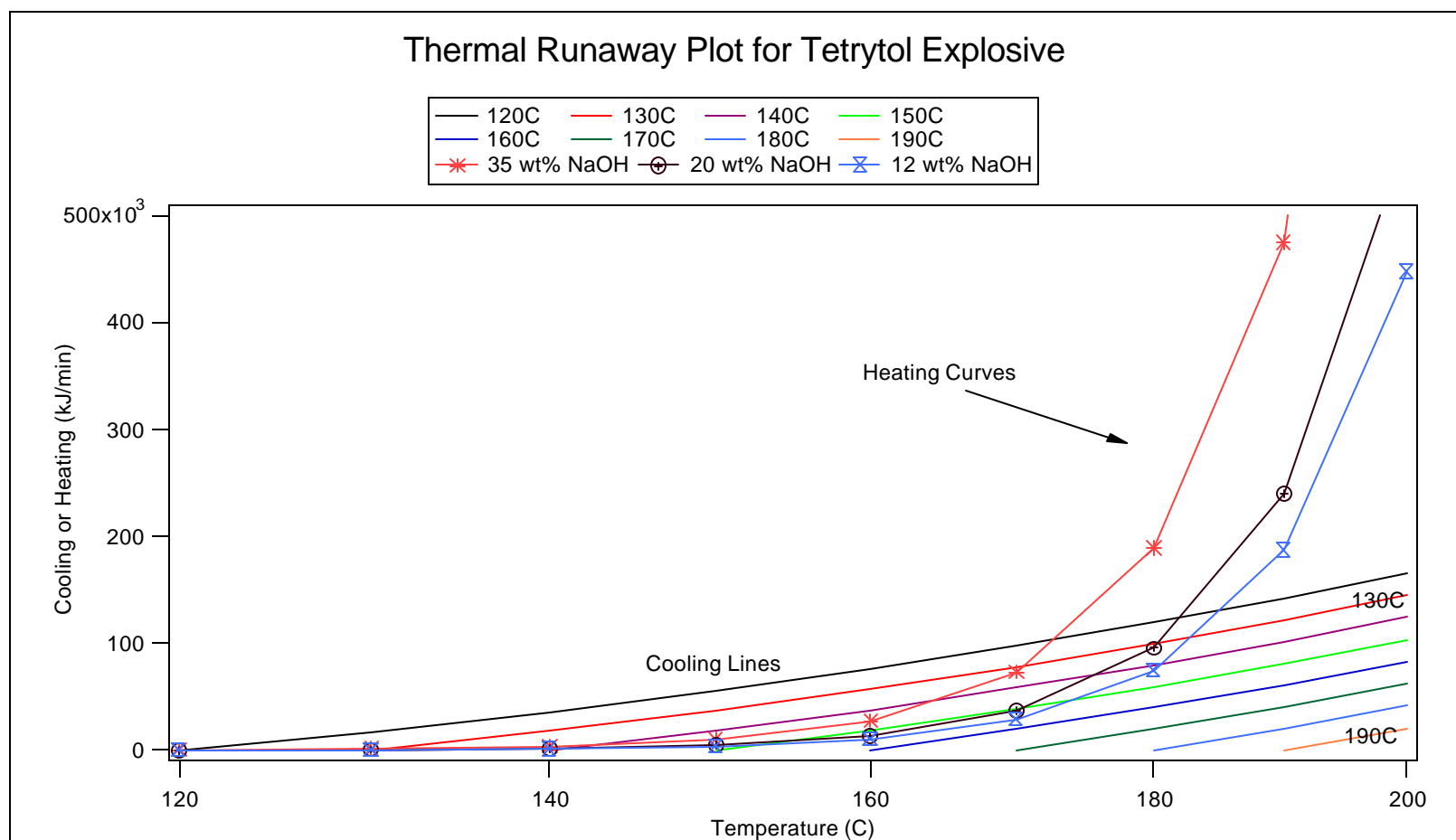
12, 20 and 35 wt% NaOH, 950 g Explosive, 1000 gal of base, Two-84 cm agitator Turning at 800 RPM.



Thermal Runaway at 150°C for 35 wt% NaOH, and 160°C for 20 wt% NaOH, and 12 wt% NaOH

#### 4.2.5 Tetrytol

12, 20 and 35 wt% NaOH, 950 g Explosive, 1000 gal of base, Two-84 cm agitator Turning at 800 RPM.



Thermal Runaway at 150°C for 35 wt% NaOH, 160°C for 20 wt% NaOH and 12 wt% NaOH

## 5.0 Discussion

The heat of reaction was measured using a simple, small-scale, differential-thermal-analysis method. The results using this method had a large amount of variation between samples, and in some cases were difficult to interpret due to foaming and/or boiling problems. However, this data was found useful for a first approximation of the heat liberated during the base hydrolysis reaction. The heat of reaction information, along with previous reaction rate and product information was integrated into a Mathcad<sup>TM</sup> program to predict products and heat produced during a large scale hydrolysis run. The information should be used to aid in the scale-up and design of future reactors. Finally, base hydrolysis data obtained from these two studies was used to determine the thermal runaway temperature threshold for all five explosive and propellants studied. The thermal runaway calculations show that there should be no safety problems if the hydrolysis reactions are run below 130°C. This is well above any temperatures postulated for any atmospheric reactor design.

## 6.0 Conclusions

- 1) Hydrolysis of M1, M8, M28, Tetrytol, and Composition B-4 is an effective and safe method for the demilitarization of high explosives.
- 2) The thermal runaway temperature for the propellants and explosives studied are 130°C or above for all base concentrations (12 – 35 wt%) studied.
- 3) The base hydrolysis of M8 propellant using 12 wt% NaOH resulted in the base solutions boiling over during the run. This should not be a problem in a larger reactor where cooling water is available to control the temperature.
- 4) The base hydrolysis of Composition B-4 using 20 and 35 wt% NaOH resulted in some of the flasks foaming over.
- 5) The base hydrolysis of Tetrytol using 35 wt% NaOH resulted in all of the flasks foaming over.
- 6) There was some un-reacted propellant remaining after the base hydrolysis of M1 propellant using 12 wt% NaOH.
- 7) There were bright orange flakes remaining after the base hydrolysis of M28 propellant using 20 and 35 wt% NaOH.
- 8) There was some tan powder remaining after the base hydrolysis of M8 propellant using 20 and 35 wt% NaOH.
- 9) There were no explosions or violent reaction of the explosives or propellants during any of the experiments to date.

## 7.0 Recommendations

A more complete thermal analysis of the base hydrolysis of the propellant and explosives needs to be done along with a more detailed modeling effort. This would take considerable time and effort. Safety experiments on a larger scale would also be useful.

## **8.0 Acknowledgments**

Los Alamos National Laboratory would like to acknowledge the PMACWA program for funding this research, specifically Scott Sussman, Bishara Elamasri and Peter Bonnet. Special thanks to Jose' Archuleta, Dale Counce, Gregg Sullivan, Ken Laintz, Sheldon Larson, Ray Flesner and John Sanchez for all their help.

## **APPENDIX E**

### **Radford AAP M28 Surrogate Propellant/Hydrolysate Scope of Work**

## **SECTION C STATEMENT OF WORK**

### **MANUFACTURE OF M28 SURROGATE PROPELLANT & HYDROLYSATES**

#### **C.1.1 SCOPE**

The contractor shall manufacture M28 surrogate propellant and produce hydrolysates from the M28 surrogate propellant, as well as process and analytical data, and an evaluation of procedures with respect to safety and efficacy.

#### **C.1.2 BACKGROUND**

In accordance with Public Law 104-208, the Under Secretary of Defense for Acquisition and Technology has appointed a Program Manager for Assembled Chemical Weapon Assessment (PM ACWA) with the mission to demonstrate at least two alternate technologies to incineration for the disposal of assembled chemical weapons (CW). Assembled CW for this purpose are representative of the CW stockpile; i.e., CW configured with fuzes, explosives, propellant, chemical agents, shipping and firing tubes, and packaging materials. Six technologies have been selected to proceed into the demonstration phase under this program, three of which incorporate the hydrolysis of energetics as the deactivation mechanism. Hydrolysate produced from M28 surrogate propellant will be generated and provided to these selected contractors as GFM to serve as hydrolysate feedstock to evaluate and validate these processes.

#### **C.1.3. SPECIAL CONSIDERATIONS**

The Government requires support that is independent and conflict-of-interest-free; unbiased review, evaluation, and analysis of hydrolysis generation procedures developed by contractors. The nature of this effort requires that the contractor work closely and effectively with all PM ACWA program contractors and their subcontractors.

## **C.2. APPLICABLE DOCUMENTS -- ANSI/ASQC E4**

## **C.3. REQUIREMENTS**

C.3.1. The contractor, as an independent contractor and not as an agent of the Government, shall furnish the necessary resources (except for those delineated as Government furnished property, equipment, or assistance) to accomplish the effort set forth below.

**C.3.2. M28 Surrogate Propellant.** The contractor shall produce 1,000 lb. of M28 surrogate propellant for subsequent use in the preparation of the M28 hydrolysates per the following requirements:

C.3.2.1 Composition: The M28 surrogate propellant will meet the nominal composition requirements for M28 propellant set forth below. However, unlike the original M28 propellant, the surrogate will be pressed, not cast, with a greater than 95% TMD. In addition, the Lead Stearate shall be eliminated from the composition.

Nitrocellulose	60.0 %
Nitroglycerin	23.8 %
Triacetin	9.9 %
Dimethylphthalate	2.6 %
Lead Stearate ( <b>delete</b> )	2.0 %
2-Nitrodiphenylamine (2-NDPA)	0.7 %

The propellant grains shall be coated with 0.1% graphite to mitigate electrostatic discharge (ESD) concerns.

**C.3.2.2** Residual solvent levels: Less than 1%.

**C.3.2.3** Grain Geometry: Size AHH:

Diameter: 0.070" nominal  
Length: 0.070" nominal

Note: This configuration can be produced using existing, qualified double-base dies.

**C.3.2.4** Ballistics: There are no ballistic requirements for the M28 surrogate propellant produced under this contract.

**C.3.2.5.** Manufacturing Plan. The contractor shall prepare and submit an M28 Surrogate Propellant Manufacturing Plan as part of the Contractor Summary Report (see paragraph C.3.5.) that is based on and represents the specific needs of the

ACWA program as provided by the Government. This Plan shall be used to produce the M28 surrogate propellant in a safe and efficient manner. The Plan shall include monitoring procedures that will be used during the manufacturing process to effectively record the process parameters and controls. The plan shall be submitted as a separate section to the contract summary report.

**C.3.2.6.** Analytical and QA/QC Plan. The contractor shall use existing QA/QC plans, modified as required, to monitor and control the manufacture of the M28 surrogate propellant.

**C.3.2.7.** Testing Requirements. Testing shall be conducted as necessary to revise the manufacturing procedures and to confirm that the compositional requirements for the M28 surrogate propellant are met. Test results shall be included in the Contractor Summary Report.

### **C.3.3. M28 Surrogate Propellant Hydrolysate.**

C.3.3.1. Hydrolysate Generation Plan: The contractor shall prepare and submit a Hydrolysate Generation Plan in contractor format DF-ADMN-80447\* tailored. This Plan shall be used to generate the M28 surrogate propellant hydrolysates in a safe and efficient manner. The Plan shall include the following information that will be used to effectively control and record the hydrolysis process:

- 1) Equipment requirements
- 2) Material handling procedures
- 3) Processing procedures and parameters
- 4) Instrumentation and monitoring controls
- 5) Safety controls
- 6) Handling of gaseous effluents and solid residuals waste
- 7) Packaging configuration for shipment

C.3.3.2. Analytical and QA/QC Plan. The contractor shall prepare and submit in contractor format an Analytical and QA/QC Plan (DF-ADMN-80447\* tailored) that reflects the requirements of the Analytical Test Matrix provided by the Government (Table 1, paragraph C.3.3.3.). This Plan shall include the sampling and analysis methods and quality assurance/quality control procedures that will be used to control



and monitor the generation of the hydrolysate, specifically: characterization of the liquid hydrolysate and the gaseous effluents and solid residuals produced by the hydrolysis process. Guidelines for preparing the QA/QC program can be found in ANSI/ASQC E4.

C.3.3.3. The following analytes shall be sampled and analyzed, as appropriate to this effort.

**Table 1. Analytical Test Matrix**

Sampling Location	Analyte	Analytical Method
<b>Reaction Vessel - Liquid</b>	Nitroglycerin Nitrocellulose, Triacetin, DMP, NDPA Metals Nitrate/Nitrite Cyanide Alcohols Carbonyl Compounds Total Organic Carbon Volatile Organics Semi-Volatile Organics Total Kjeldahl Nitrogen	SW-846 8332 TBD SW-846 6010 SW-846 9056 SW-846 9010 SW-846 8100 SW-846 8315 SW-846 9060 SW-846 8260 SW-846 8270 EPA water and waste 351.2
<b>Gaseous Outlet</b>	Nitrogen Ammonia, Nitrous Oxide Oxides of Nitrogen Nitroglycerin Nitrocellulose, Triacetin, DMP, NDPA VOCs	GC-TCD FTIR Chemilumin. Analyzer SW-846 8332 TBD SW-846 0030/5041A
<b>Solid Residue</b>	Anions (Nitrate/Nitrite) Metals, Total Semi-Volatile Organics Nitroglycerin Nitrocellulose, Triacetin, DMP, NDPA Cyanide Total Kjeldahl Nitrogen Total Organic Carbon	SW-846 9056 SW-846 6010 SW-846 8270 SW-846 8332 TBD SW-846 9010 EPA water and waste 351.2 SW-846 9060

C.3.3.4. Hydrolysate Generation: The contractor shall generate M28 surrogate propellant hydrolysates using sodium hydroxide (NaOH) solutions and the M28 surrogate propellant produced per paragraph C.3.2 above.

C.3.3.4.1. Testing Requirements. Preliminary testing (bench-scale) shall be conducted, as necessary, to develop the hydrolysate generation procedures for hydrolysis of the M28 surrogate propellant to support development of the Hydrolysate Generation Plan.

C.3.3.4.2. M28 surrogate propellant hydrolysates using two different NaOH solution concentrations:

1) Hydrolysate Number 1:

1068 pounds of NaOH in 15844 pounds water and 890 pounds M28 (w/o lead stearate) The hydrolysate shall be heated to 90°C for 12 hours. If the resulting pH is above 11, adjust the pH of the hydrolysate with sulfuric acid to achieve a pH of ~10.

2) Hydrolysate Number 2:

413 pounds of 12% NaOH and 83 pounds M28 (w/o lead stearate). The hydrolysate shall be heated to 95°C for 8 hours. Adjust the pH of the hydrolysate to a pH of ~7 using phosphoric acid

C.3.3.4.3. Process Parameters: The contractor shall record process parameters to include concentrations, loadings, temperatures, pressures, etc. per the Hydrolysate Generation Plan to generate data to be used to evaluate the processes for efficiency, efficacy and safety.

C.3.3.4.4. Analytical Testing: The contractor shall conduct analytical testing referring to the Analytical and QA/QC Plan submitted for each hydrolysate generated.

C.3.3.5. Lead Stearate Hydrolysate: The contractor shall separately prepare a Lead Stearate hydrolysate (or acceptable surrogate) that can be added at the test site to the two, M28 surrogate propellant hydrolysates produced per the requirements set forth in paragraph C.3.3.4.2.

C.3.3.5.1. The addition method for adding the Lead Stearate hydrolysate to the two, M28 surrogate propellant hydrolysates shall be fully defined to ensure that the resultant mixture will be compositionally representative to a hydrolysate produced from M28 surrogate propellant containing Lead Stearate.

C.3.3.5.2. The required quantity of Lead Stearate hydrolysates required to adjust the two, M28 surrogate propellant hydrolysates, will be shipped to the respective test sites identified in paragraph C.4.

C.3.4. Preparation for Hydrolysate for Shipment. The contractor shall prepare all hydrolysate in such a manner as to minimize the further hydrolysis of the hydrolysate after the original generation procedure.

C.3.4.1. Hazards Classification: The contractor shall be responsible for obtaining the required DOT hazards classifications and packaging configuration for the shipment of the hydrolysates to the designated sites.

C.3.5. Contract Summary Report. The contractor shall provide a Contract Summary Report in contractor format (DI-ADMN-80447\* tailored) that includes all data generated during the period of performance. The Contractor Summary Report shall include:

- 1) Independent analysis of potential safety risks and hazards associated with the procedures used to generate the hydrolysate from the M28 surrogate propellant.
- 2) Proposed process modifications (equipment, procedures, etc.) that are anticipated in order to recover from and hydrolyze the actual M28 propellant used on the 115-mm rocket (M55) and warhead (M56).
- 3) Best approach for re-introducing the hydrolysis products of lead stearate back into the feed.
- 4) Indication of amount of solids to expect in the hydrolysates.
- 5) Estimate of the scale up requirements to transition the piloted process to full scale production applications.
- 6) 'Lessons Learned' from this task.

C.4. Items to be Delivered. The contractor shall provide hydrolysate prepared IAW the requirements set-forth in paragraph C.3.3.4.2. to Dugway Proving Ground and Deseret Chemical Depot (CAMDS) by 1 January 1999 in the following quantities:

C.4.1.1 Deseret Chemical Depot (CAMDS) – Hydrolysate Number 1:

Shipping address: Deseret Chemical Depot  
Chemical Agent Munition Disposal System  
Bldg 5120  
ATTN: Joe Stilnovich  
Tooele UT 84074

C.4.1.2 Dugway Proving Ground – Hydrolysate Number 2:

Shipping address: Commander  
Dugway Proving Ground  
ATTN: Andrew Neafsey  
Dugway Proving Ground UT

C.5. Period of Performance. The period of performance for this effort will be six months from the date of contract award.

## **APPENDIX F**

### **Radford AAP M28 Propellant Incident Report**

# *Demilitarization of M28 Propellant*

## *October 14, 2000 Incident Report*

*ARDEC Contract DAAE30-97-D-1013 Task 0010*



*October 31, 2000*

Malcolm Williams

Earl Lemon

Alliant Techsystems, Inc.

# *Overview*



- Under contract to U.S. Army TACOM-ARDEC, Alliant Techsystems was tasked with manufacturing M28 surrogate propellant and then digesting this material in a caustic solution to produce hydrolysates for subsequent off plant analysis.
- An over-pressurization incident occurred during the digestion process.
  - A pump blew apart between 9:40 AM and 10:00 AM on 10/14/00 while recirculating the hydrolysate solution.
  - Secondary incident occurred in the piping system at 11:05 a.m. and again at 11:35 a.m.

# *Contractual Scope of Work*

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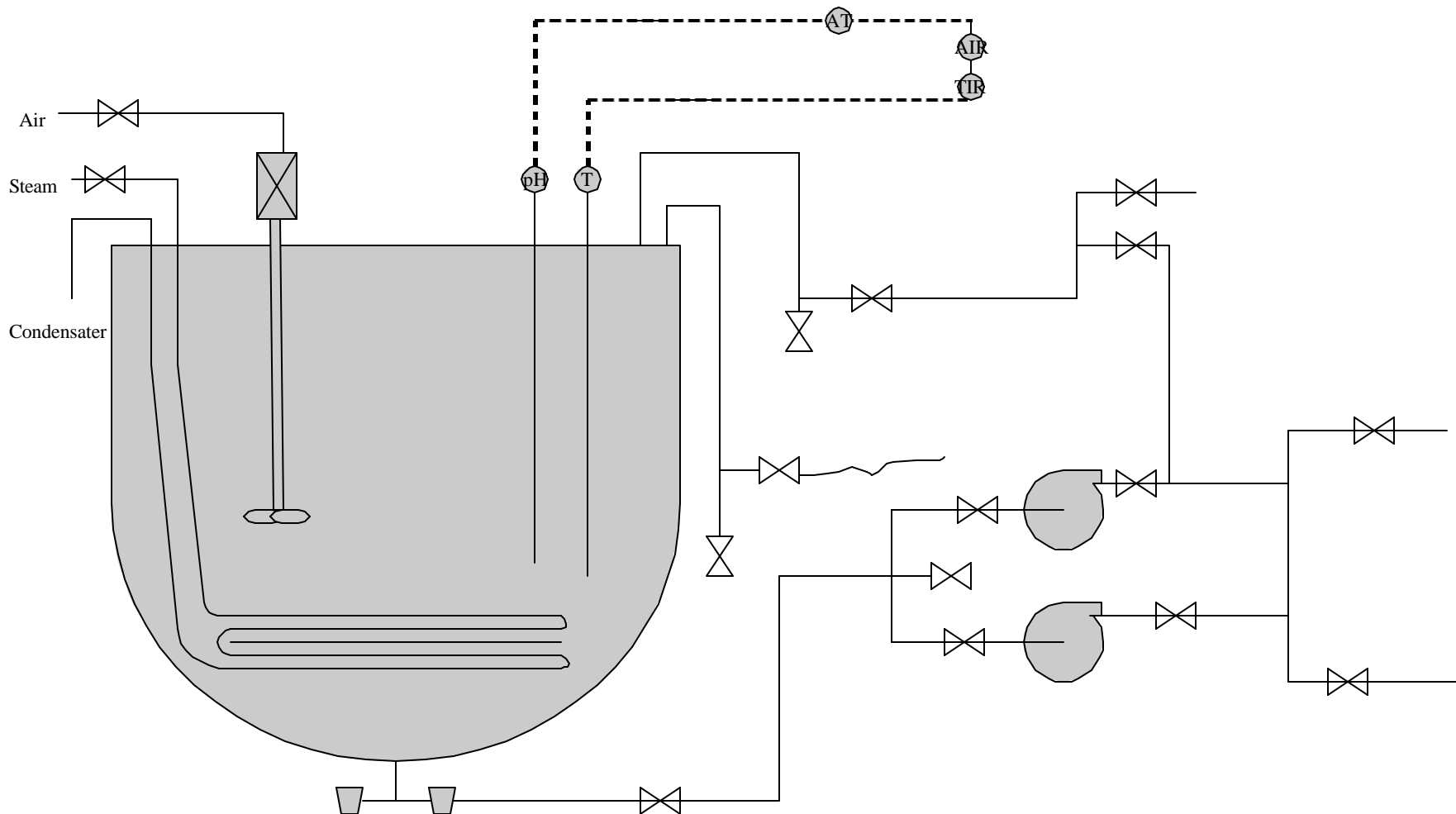
Task 1: Manufacture 2166 lbs. of M28  
surrogate propellant

Task 2: Digest the Surrogate propellant in  
9498 lbs. of 12% NaOH solution

Task 3: Neutralize solution with phosphoric  
acid for off plant shipment in 55  
gallon drums



# *Equipment Diagram*

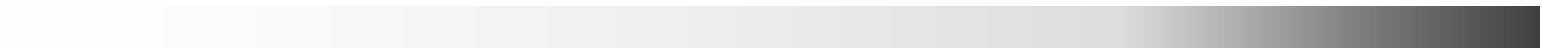


# *Chain of Events*

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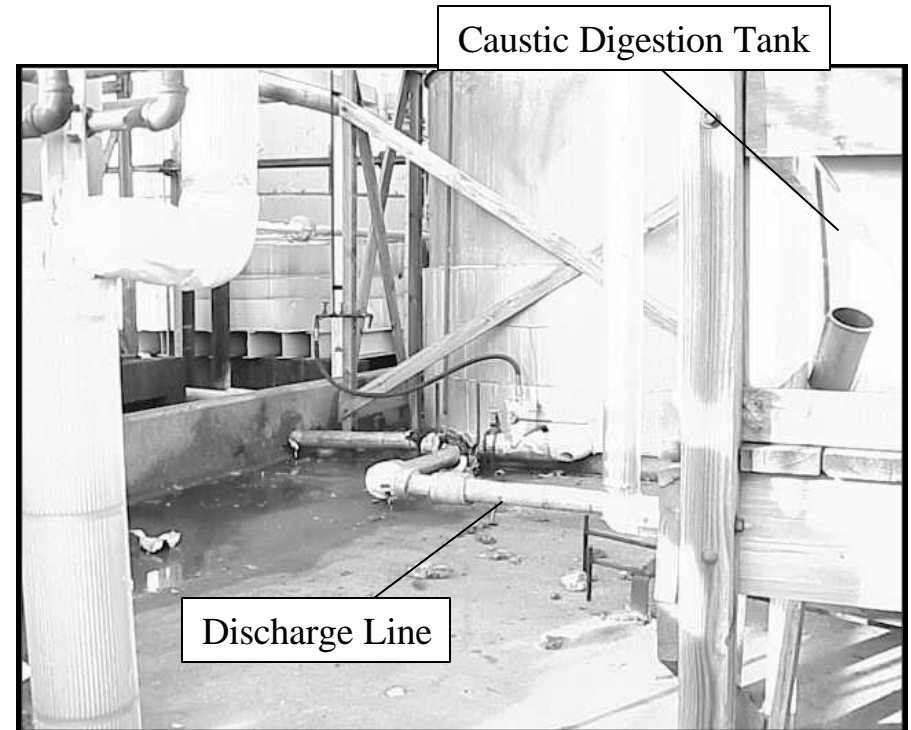
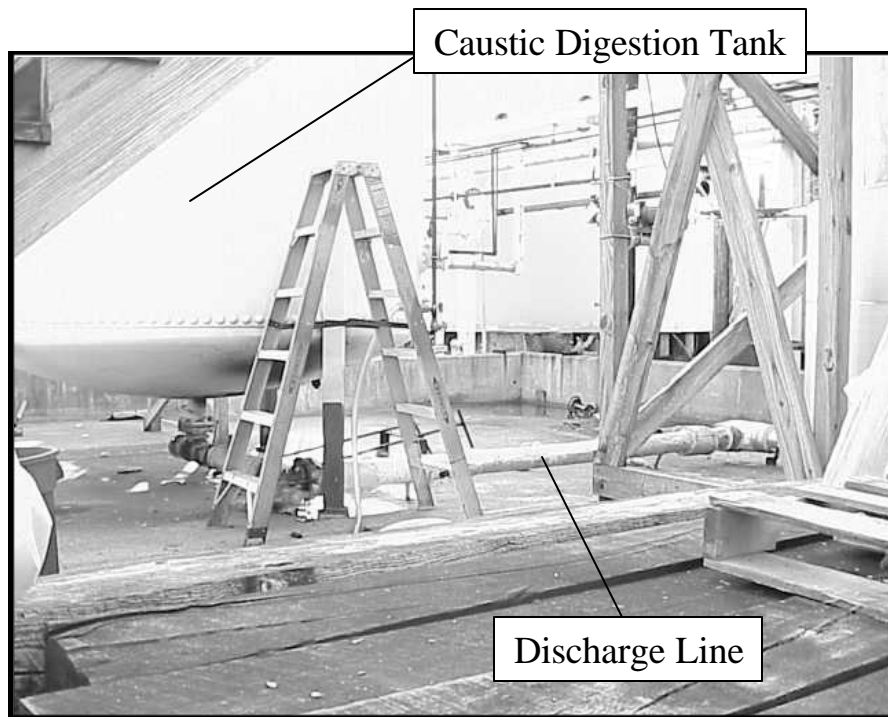
- Thursday, October 12, 2000
  - 1:00 PM Water was added to tank
  - 5:50 PM Sodium Hydroxide added to tank
  - 6:00 PM Steam Turned On
- Friday, October 13, 2000
  - 11:15 AM Began M28 propellant adds.
  - 4:15 PM Propellant adds completed
- Saturday, October 14, 2000
  - 8:15 AM West pump turned on. Pump stalled and was shut down
  - 8:25 AM East pump turned on.
  - 9:50 AM East pump found blown apart.
  - 11:05 AM Second Event Occurred.(Piping)
  - 11:30 AM Third Event Occurred ( Piping)

# *Summary of Incident*

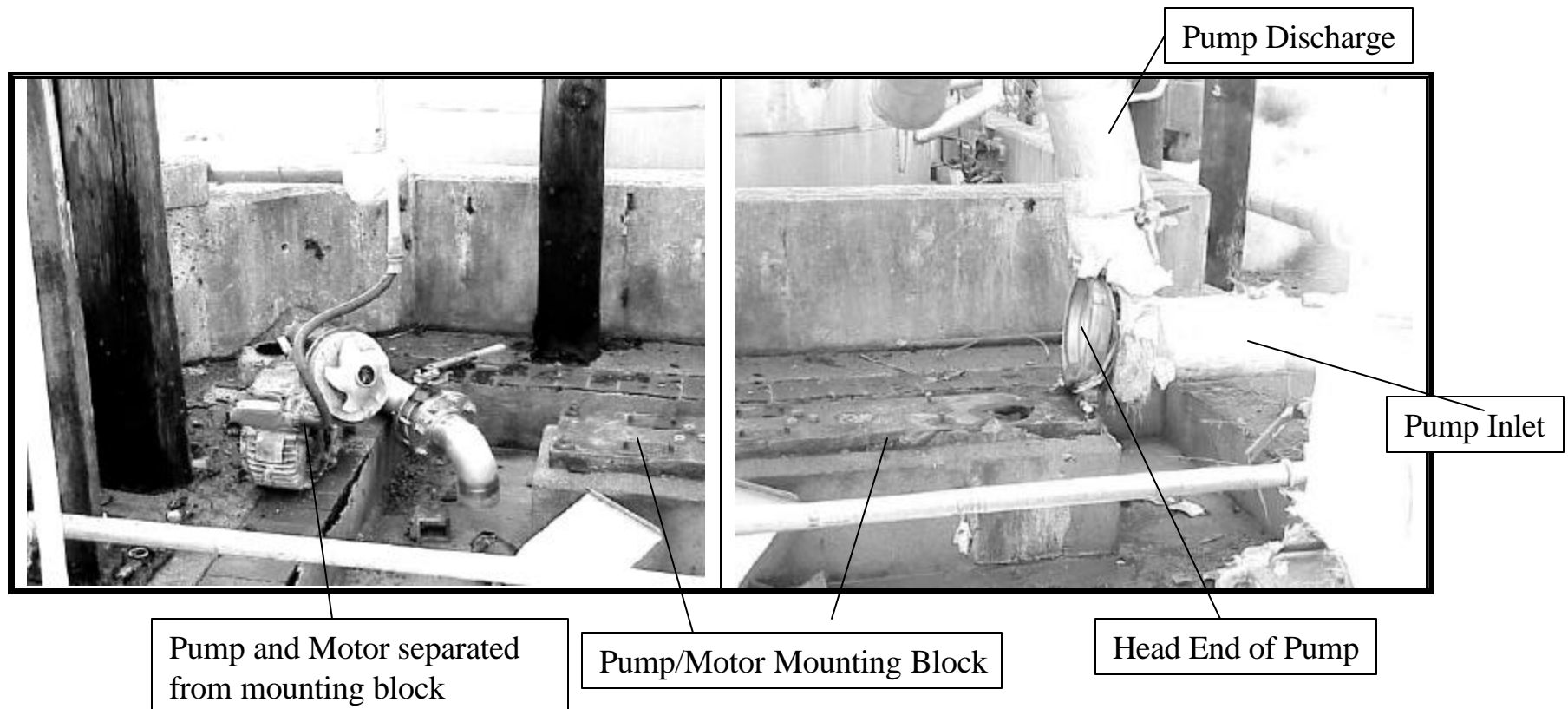


- Incident was related to over-pressurization in the piping and pump equipment.
  - Pump was pushed apart forcefully (over-pressure).
  - Subsequent pipe ruptures split and broke piping but did not fragment.
  - Unburned (partially digested) propellant found vented from piping.
- Incident was not due to propellant initiation.
  - No evidence of propellant burning was found.

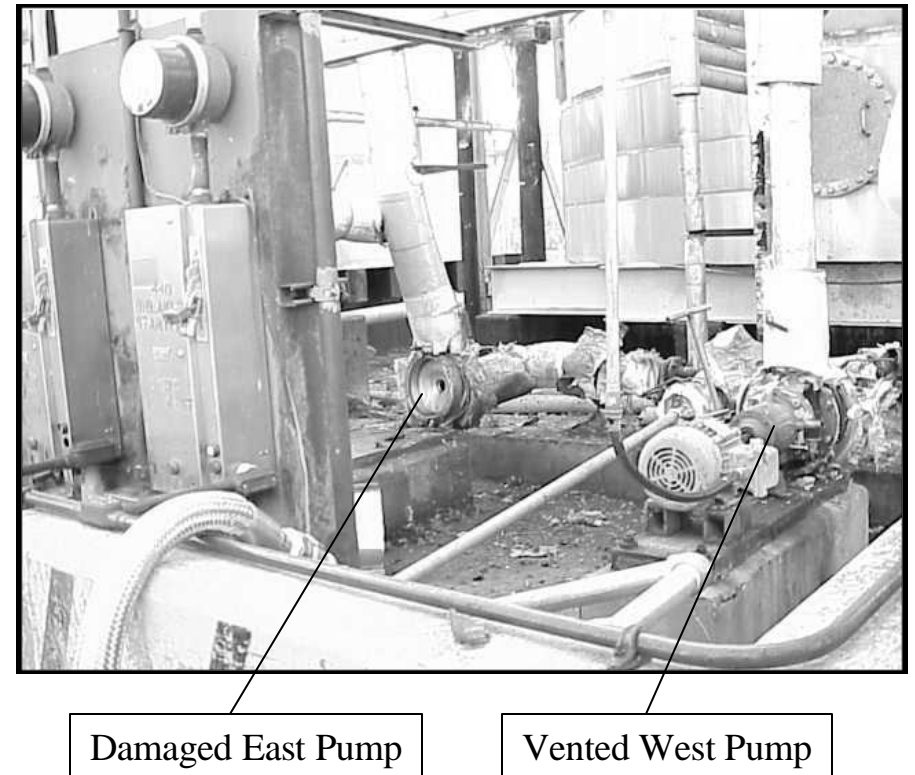
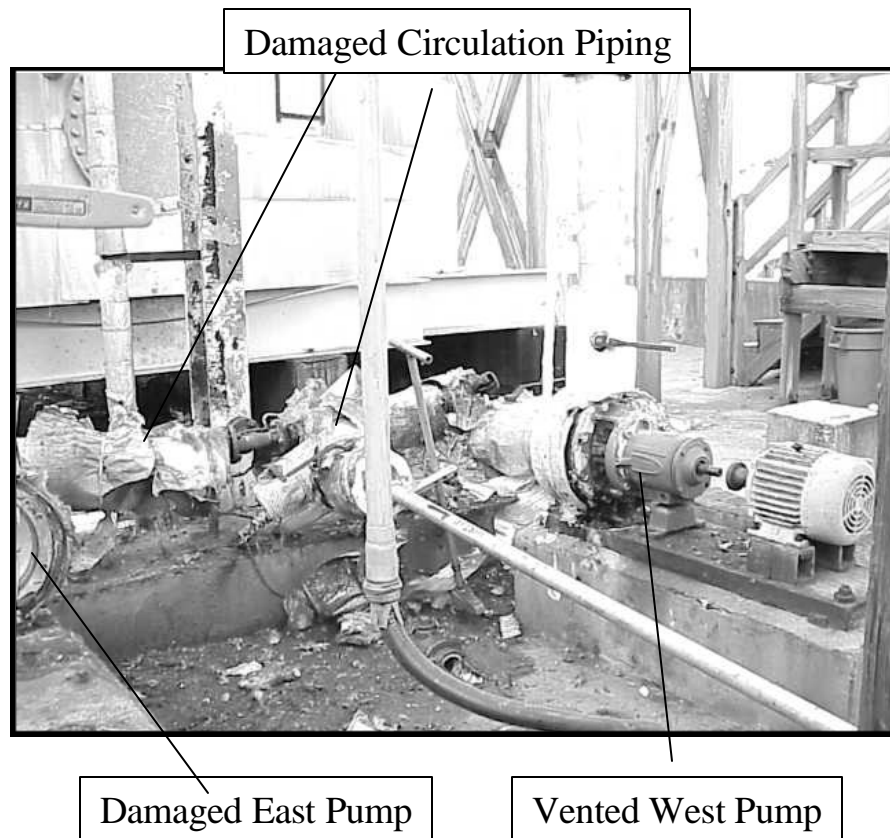
# *Photographs Show Important Aspects of the Facility Design*



# *Post-Event Photographs of Pumps Show Limited Equipment Damage*



# *Post-Event Photographs Show Minor Secondary Damage to the Recirculation System*



# *Post-Event Photographs Show Minor Secondary Damage to the Re-circulation System*

Damaged East Pump

Vented West Pump

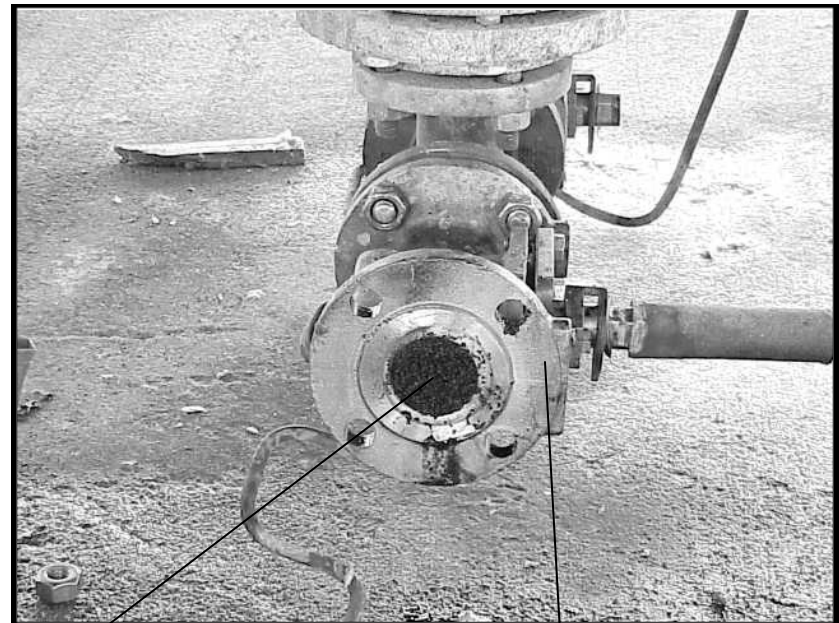
Damaged Piping



# *Undigested M28 Propellant Found Throughout the Re-circulation Piping System*



M28 Propellant



Valve Located at Base of Tank



# *Release of Undigested M28 Propellant Was Limited to a Small Area Around the Piping*



# *Investigation Quickly Identified the Incident Cause*

---

- Over-pressurization condition was created by the plugging of the suction line for the re-circulation system with undigested propellant that continued to react with the caustic solution.

# *Chain of Events Led to the Incident*

---

- Incomplete Digestion of Propellant led to,
- Re-circulation Pumps and Piping Plugged with Propellant which,
- Continued to React Due to Energy Input By Pump and Steam Trace Line Heating which,
- Created Over-pressure Conditions Within the Pump and Piping which,
- Eventually Caused the Pumps and Piping to Rupture.

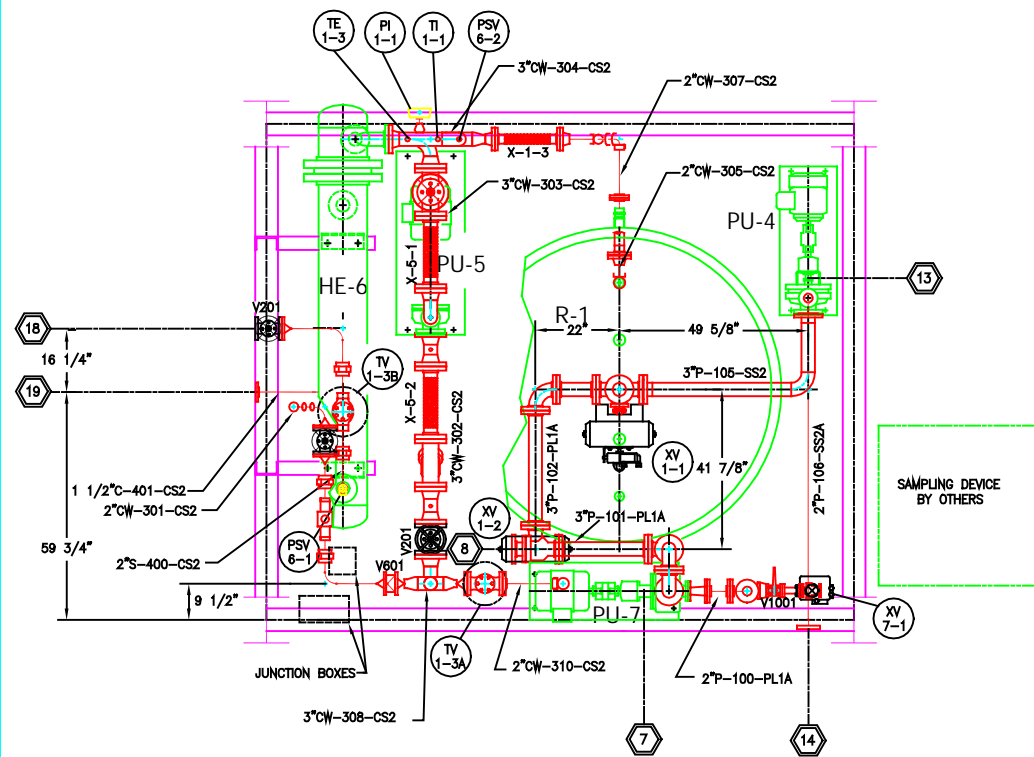
# *Lessons Learned*



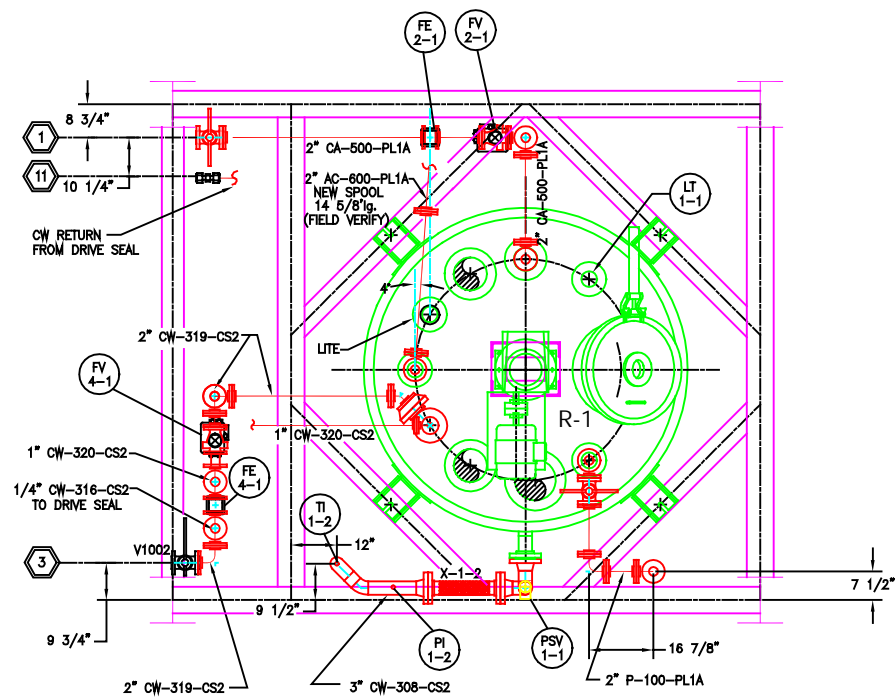
- Insure that enough reactants are present for complete reaction of all propellant.
- Assure solids cannot get into pumping system.
- Pumps should have flow interlocks.
- Assure reacting material cannot become isolated in the system.
- Identify all potential energy input sources.

## **APPENDIX G**

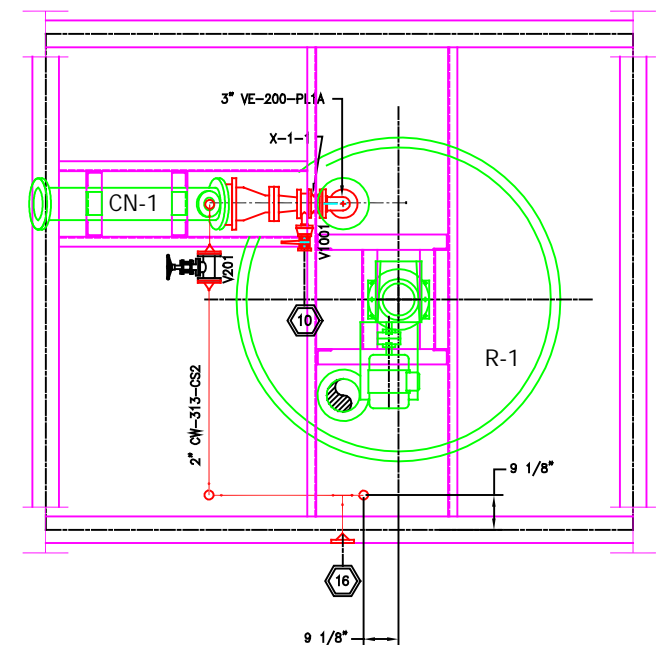
### **Pfaudler Reactor Specifications and Requirements**



EQUIPMENT PLAN @ EL. 8" T.O.S.



EQUIPMENT PLAN @ EL. 124" T.O.S.



EQUIPMENT PLAN @ EL. 204" T.O.S.



				4	10/25/00	REVISED LINE 2" AC-600-PL1A	GS
				3	10/02/00	AS BUILT CHANGES	GS
				2	9/26/00	AS BUILT CHANGES	GS
				1	7/12/00	ISSUED FOR CONSTRUCTION	GS
				0	7/7/00	ISSUED FOR REVIEW	GS
NO.	DATE	REVISIONS	BY	NO.	DATE	REVISIONS	BY

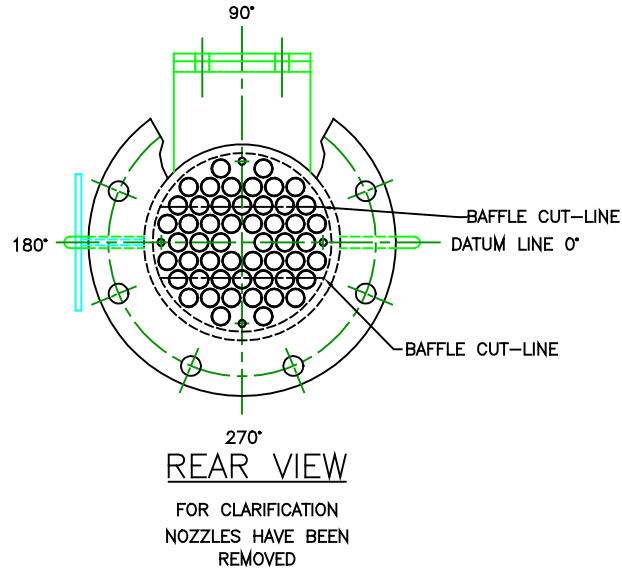
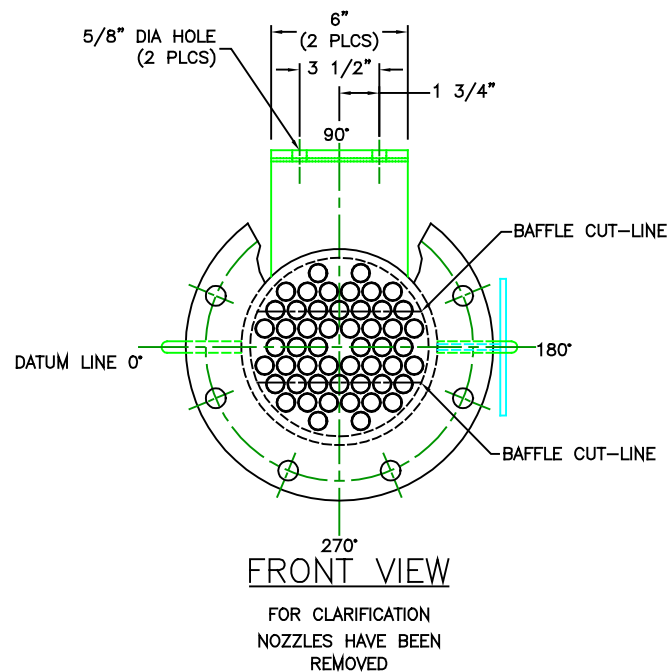
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SCALE 1=24	CHECKED	DATE
DATE 7/7/00	APPROVED	DATE
DRAWN RJD	APPROVED	DATE
WORK FROM DIMENSIONS - DO NOT SCALE		

PIPING LAYOUT - PLAN VIEWS  
2000 GAL. G/L REACTOR SYSTEM  
U.S. ARMY - PICATINNY ARSENAL, N.J.



Drawing No.  
JD-00-6251-300-4

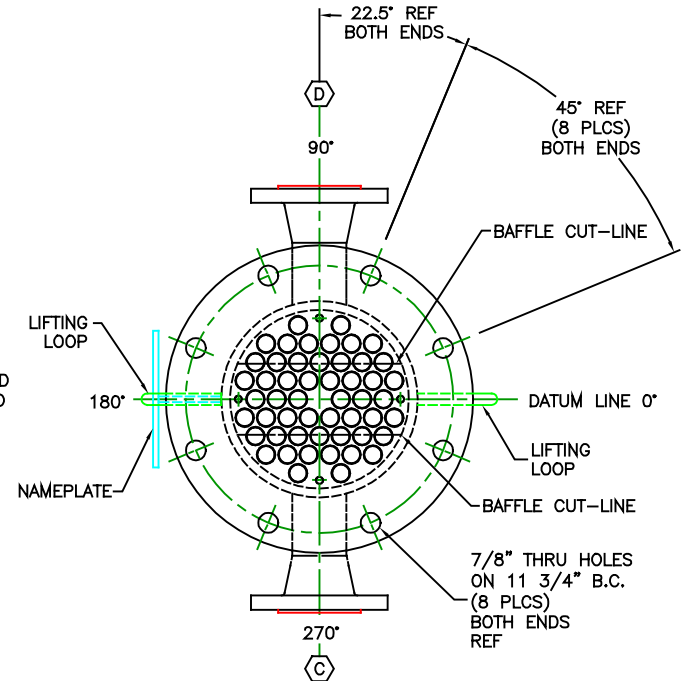
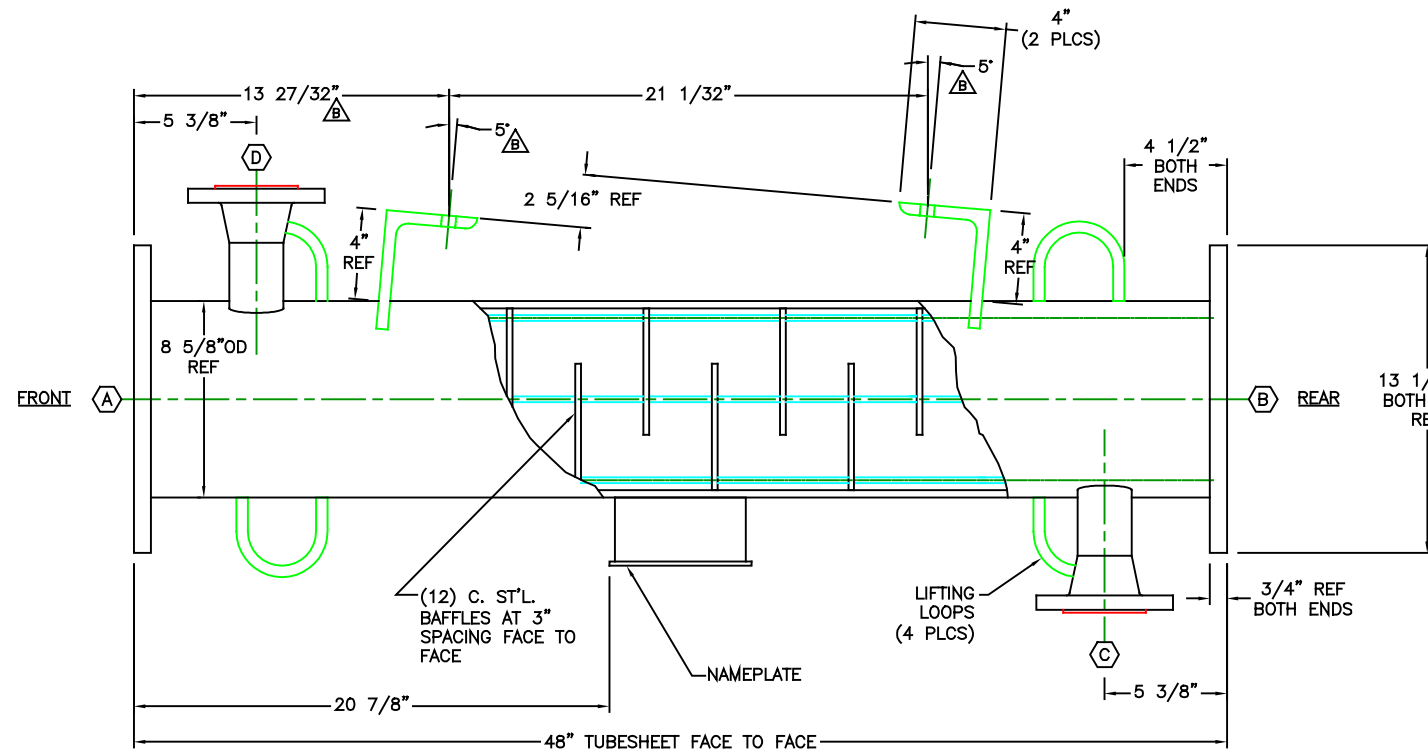
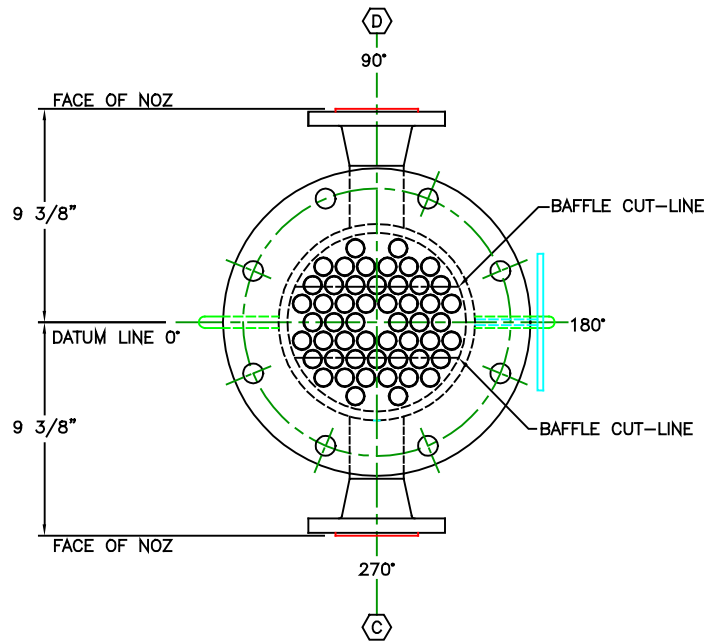


## NOTES:

- 1.) PRESSURE RELIEF DEVICE(S) PER PAR UG-125 OF ASME CODE, SECT. VIII DIV 1, MUST BE PROVIDED AND INSTALLED BY CUSTOMER.
- 2.) IN ORDER TO AVOID INJURY AND ASSURE THE SAFE OPERATION OF ALL PFAUDLER VESSELS, NO VESSEL SHOULD BE OPERATED WITHOUT ALL OPENINGS AND PASSAGEWAYS SECURED BY A SUITABLE COVER WHILE THE VESSEL IS IN OPERATION. ALL PERSONS OPERATING A PFAUDLER VESSEL SHOULD HAVE PRIOR INSTRUCTION IN THE PROPER USE OF THE VESSEL DURING OPERATION.
- 3.) ALL FLANGED NOZZLES TO BE FITTED WITH PLYWOOD COVERS FOR SHIPPING ATTACHED WITH NYLON STRAPS.
- 4.) LIQUID PENETRANT EXAMINATION OF ALL C-276 MATERIAL WELDS PER ASME CODE PARA. UNF-58(C).
- 5.) FLANGE BOLT HOLES STRADDLE CENTERLINES.

REF	SIZE	TYPE	DESCRIPTION
A	8" CL150	TUBESHEET, FLAT FACE	PROCESS INLET
B	8" CL150	TUBESHEET, FLAT FACE	PROCESS OUTLET
C	2" CL150	RF WELD NECK	COOLING FLUID INLET
D	2" CL150	RF WELD NECK	COOLING FLUID OUTLET

SEE PFAUDLER ORDER SPEC DATA SHEET FOR COMPLETE COMPONENT DESCRIPTION



VESSEL AND/OR JACKET COMPONENTS WELDED IN ACCORDANCE WITH WELD PROCEDURES IN SECTION DA-2-4 OF THE PSA & ROCHESTER WELDING MANUAL

DESIGNED IN ACCORDANCE WITH SECTION VIII DIV. 1 OF THE ASME CODE, TEMA CLASS & IF APPLICABLE, CUSTOMER SPEC'S, WITH PFAUDLER EXCEPTIONS

FOR INSTALLATION IN AREAS NOT SUBJECT TO WIND OR SEISMIC LOADS

UNLESS OTHERWISE SPEC., ALL FLANGE BOLT HOLES STRADDLE CL'S (ANSI B16.5 BOLTING)

WORK FROM DIMENSIONS - DO NOT SCALE - SEE PLAN VIEW & BOTTOM VIEW FOR ORIENTATION

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LTR	REVISION	APP	DATE
A	CHANGED BAFFLE NOTE AND NOZZLE C AND D ORIENTATION	PH	6-1-00
B	ROTATED SUPPORTS 10 DEG TO REAR NOW 5 DEG SLOPE TO REAR WAS 6 DEG TO FRONT SLOPE PER CUST ECH7177		6-12-00

UNLESS OTHERWISE SPECIFIED DIMENSIONS ARE IN INCHES

**TOLERANCES**  
FRACTIONS:  $\pm 1/4"$   
DECIMAL:  $\pm .010"$   
ANGLES:  $\pm 1^\circ$

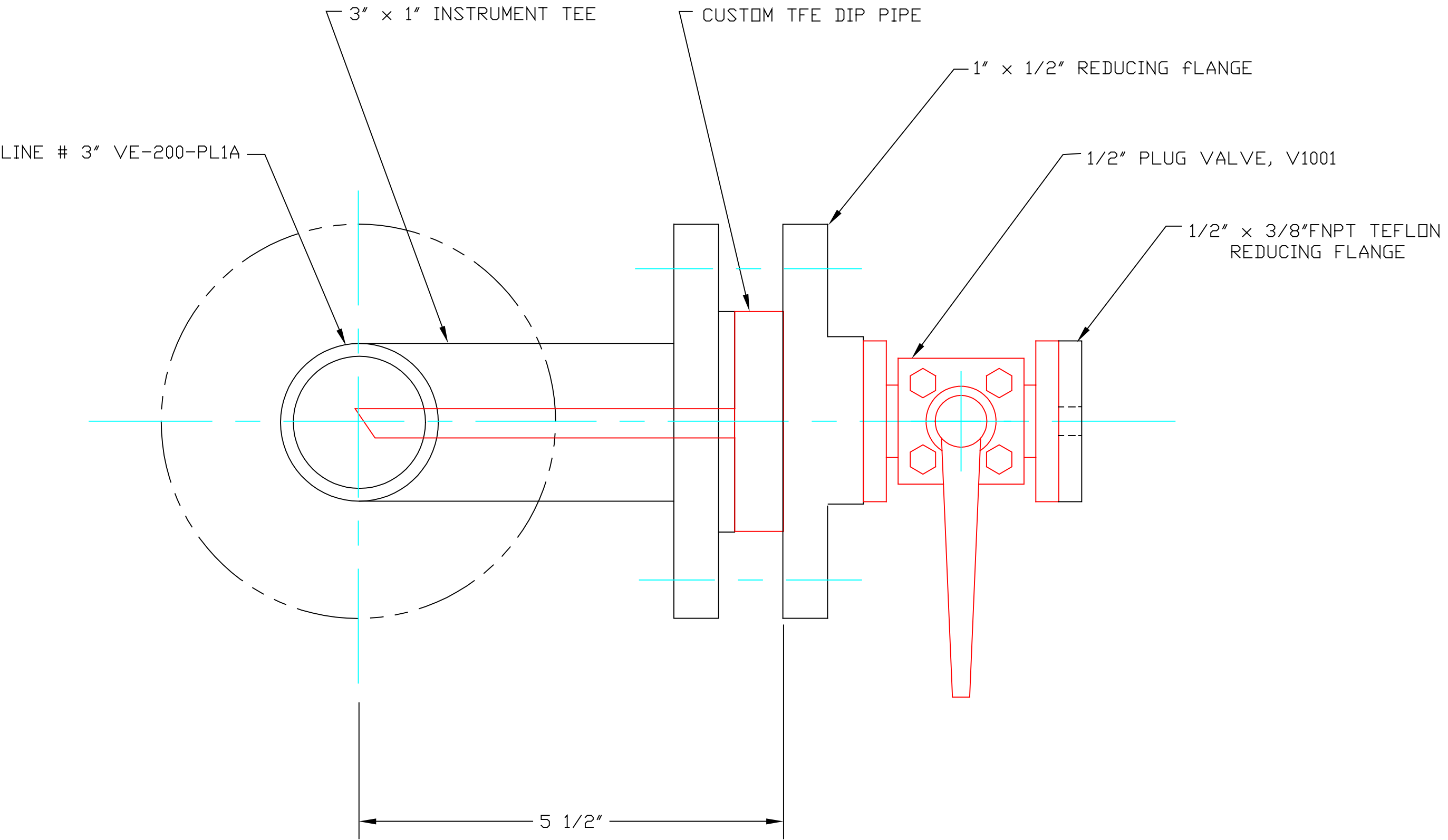
DESIGN & INSPECTION			BASE MATERIAL			VESSEL FINISH & ACCESSORY MATERIALS		
SPECIFICATION	SHELL SIDE	TUBE SIDE	SHELL:	SA-106 GR B SML		INTERNAL:	MILL FINISH, WELDS WIRE BRUSH	
ASME CODE DESIGN & STAMP	YES	YES	FRONT BONNET:	NONE		EXTERNAL:	C.S. PARTS - BLAST PER SSPC-SP-C	
DESIGN PRESS. PSI	100	100/FV	REAR BONNET:	NONE		PAINT:	STANDARD AMERLOCK EPOXY 400 BL-5 PER D.S. 61-200 (HORIZON BLUE)	
DESIGN TEMP. (MAX.) °F	350	350	TUBES/QT:	(62) 3/4" OD, SB-626 C-276, WLD		GASKETS:	NONE	
MIN DESIGN METAL TEMP °F	-20	-20	TUBESHEET:	SB-675 C276		HEATED SURFACE AREA:	40 SQ FT	SQ.FT.
HYDROTEST PRESS. PSI	130	130	THICKNESS	SHELL SIDE	TUBE SIDE	WT. BUNDLE	351 LBS.	WT. EMPTY
RADIOGRAPH	NONE	NONE	FRONT HEAD (MIN)	NONE	NONE	WT. W/H2O	435 LBS.	
CORROSION ALLOWANCE	.062"	NONE	REAR HEAD (MIN)	NONE	NONE			
NUMBER OF PASSES	1	1	SHELL (NOM)	.322"	NONE			
			TUBES (AVG)	---	.049"			

CUSTOMER: PFAUDLER, INC  
ADDRESS: ROCHESTER, NY  
CONSIGNEE: PKG EQUIPMENT, INC  
ADDRESS: ROCHESTER, NY  
CUST. ORDER NO.: G006251  
CUST. TAG NO.: CN-2

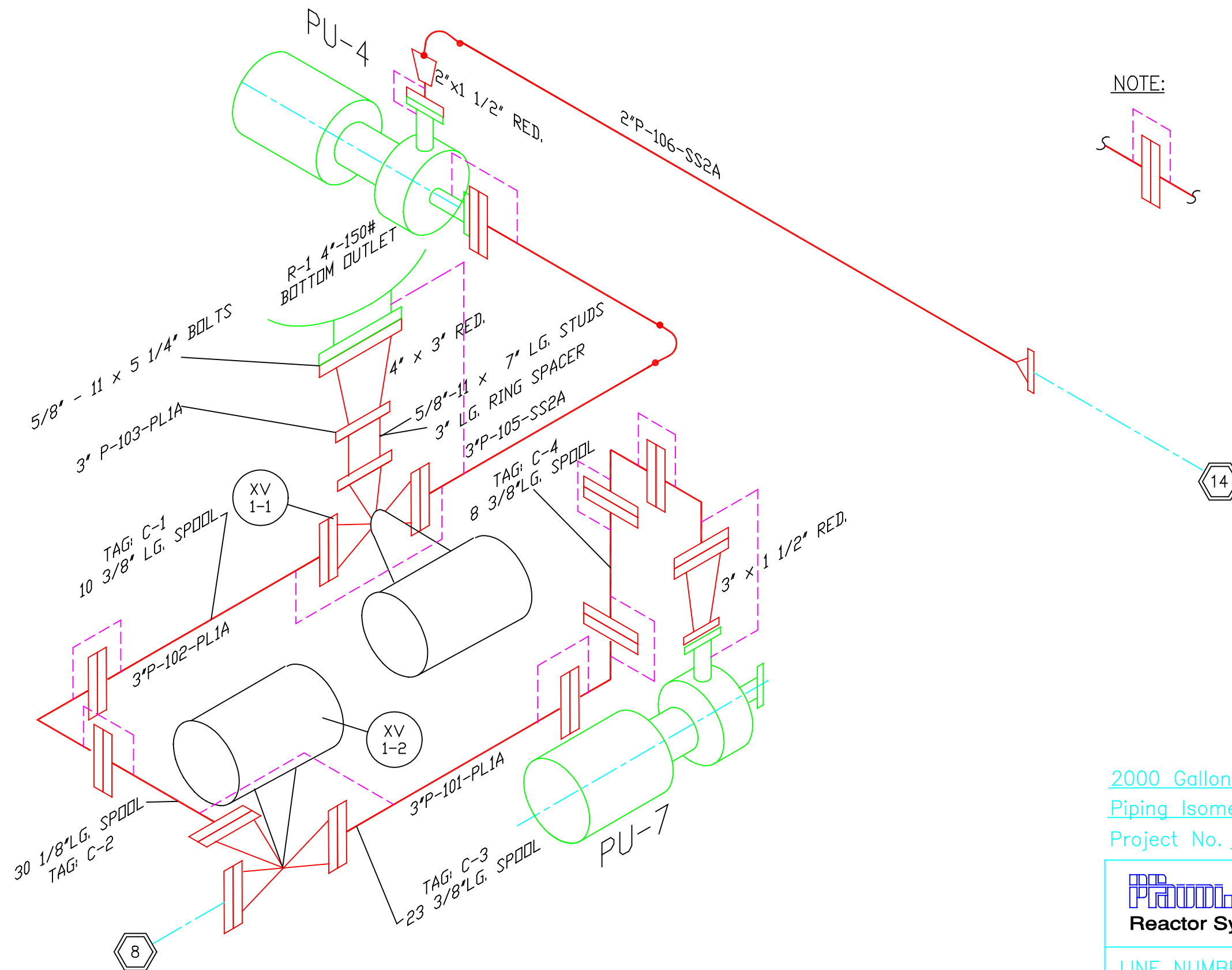
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40 SQ FT  
HORIZ., 1 PASS

**PFAUDLER**  
Pfaudler Inc. Rochester, NY

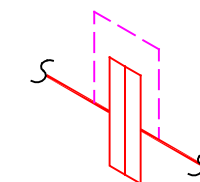
DRAWING NO. H001020  
SHEET 1  
REV B



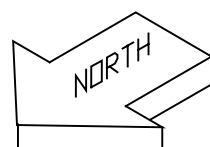




NOTE:



BONDING JUMPER, TYPICAL  
AT ALL FLD'D CONN.



2000 Gallon G/L Reactor System – U.S. Army  
Piping Isometric Diagram  
Project No. G00-6251

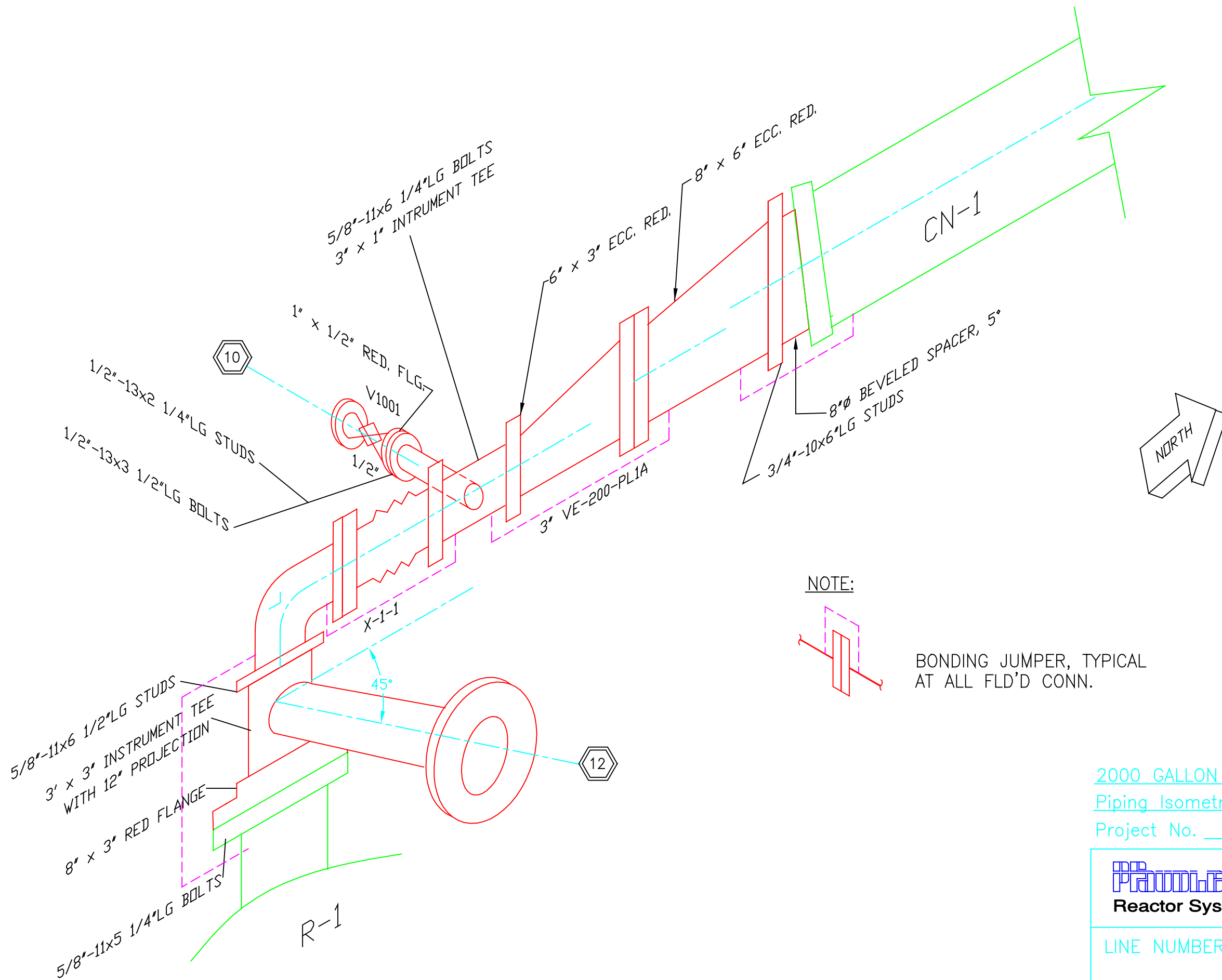
**PRANDLER, Inc.**  
Reactor Systems

REV. 7/18/00

SHT. 1 OF 9

LINE NUMBER'S : 101, 102, 105, & 106



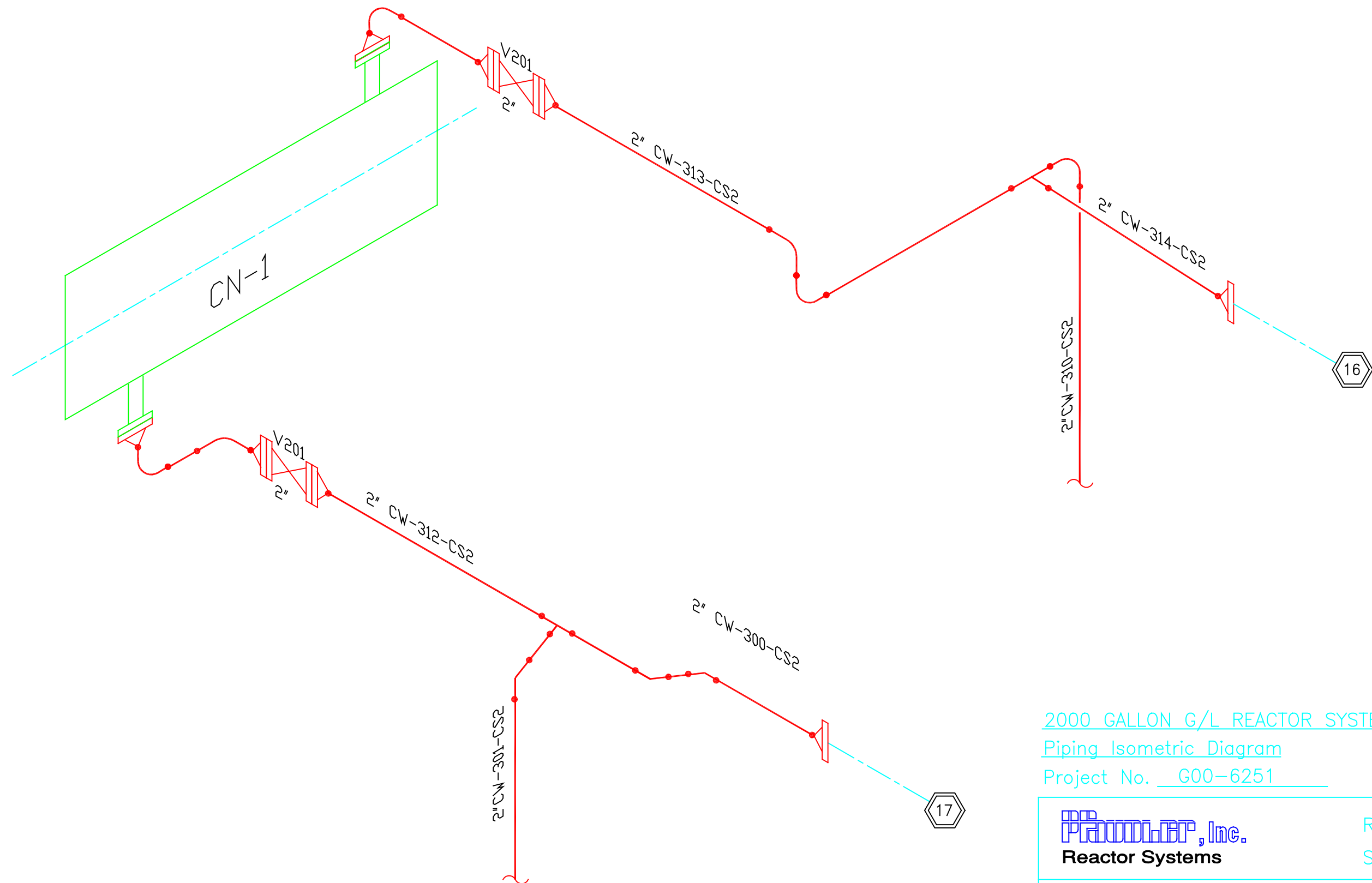


2000 GALLON G/L REACTOR SYSTEM  
Piping Isometric Diagram  
Project No. G00-6251

**PRIMET, Inc.**  
Reactor Systems

REV. \_\_\_\_\_  
SHT. 3 OF 9

LINE NUMBER'S : 200

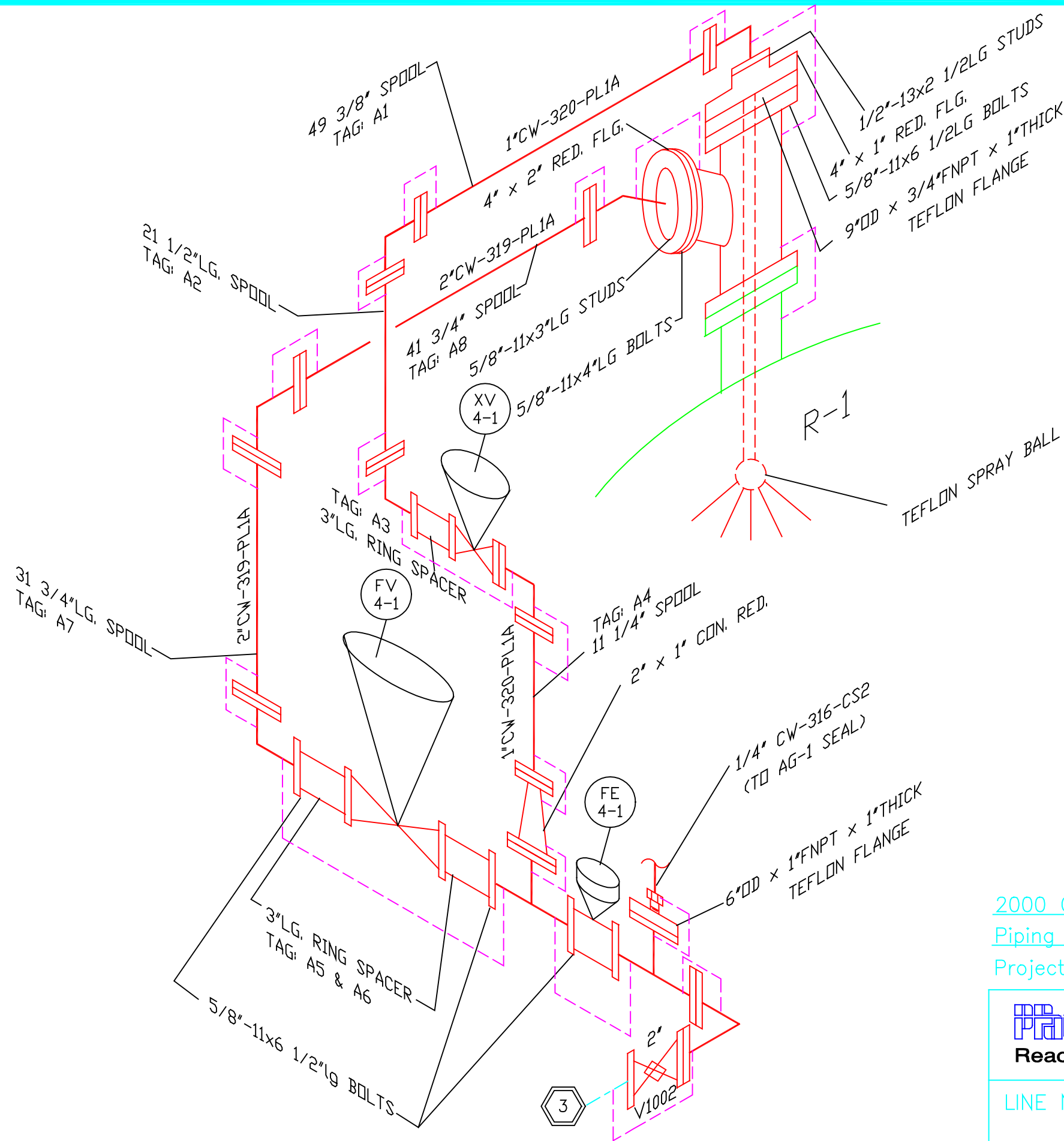
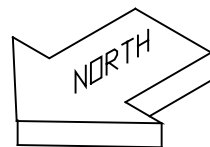


2000 GALLON G/L REACTOR SYSTEM  
Piping Isometric Diagram  
Project No. G00-6251

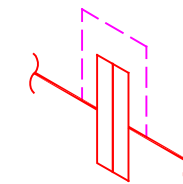
**PRANDLER, Inc.**  
Reactor Systems

REV. \_\_\_\_\_  
SHT. 4 OF 9

LINE NUMBER'S : 300, 310, 312, 313 & 314



NOTE:



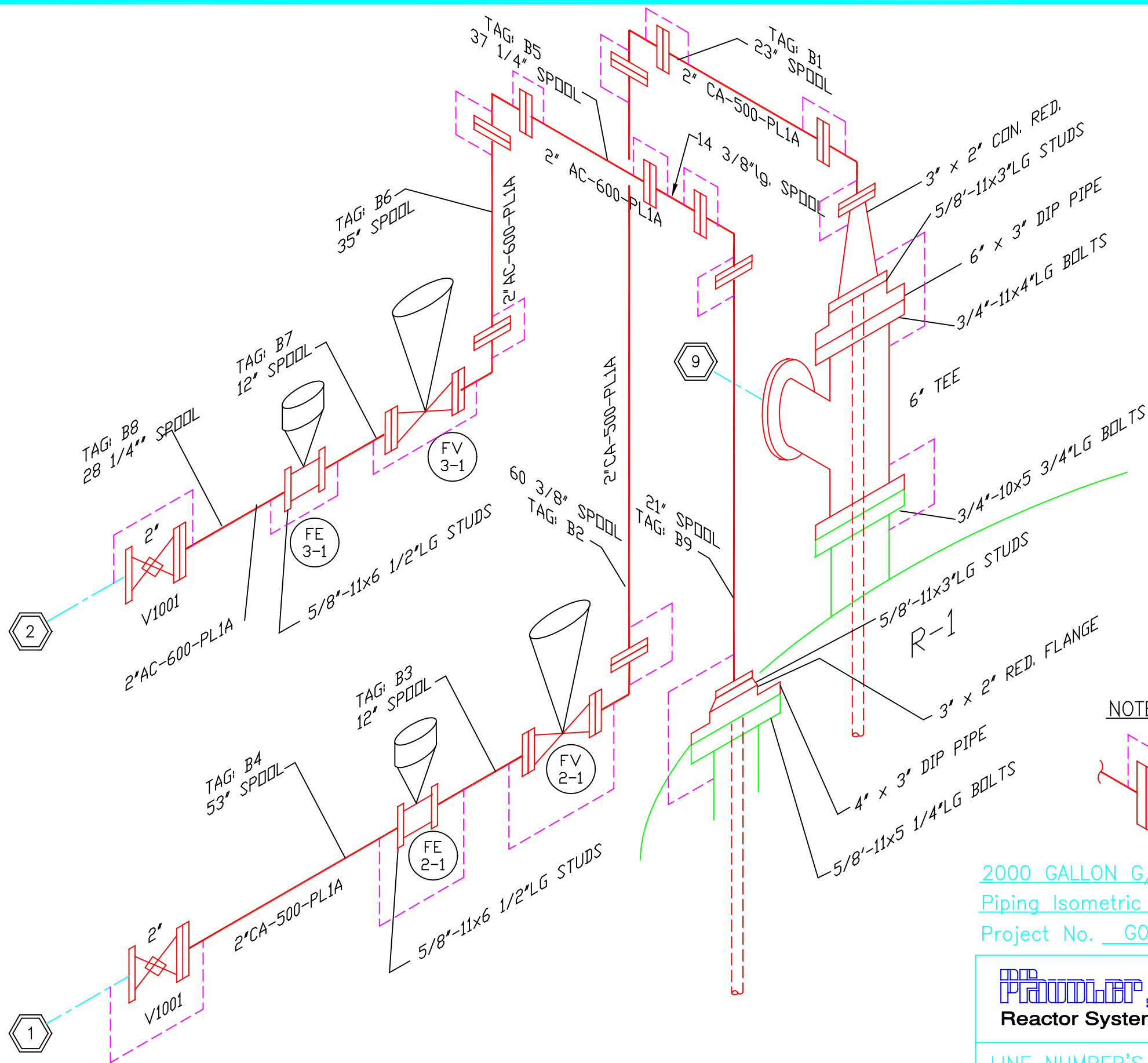
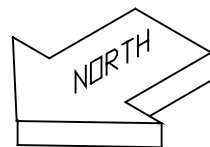
BONDING JUMPER, TYPICAL  
AT ALL FLD'D CONN.

2000 GALLON G/L REACTOR SYSTEM  
Piping Isometric Diagram  
Project No. G00-6251

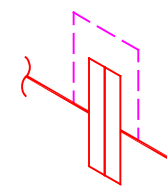
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Reactor Systems

REV. \_\_\_\_\_  
SHT. 5 OF 9

LINE NUMBER'S : 316, 319, & 320



NOTE:



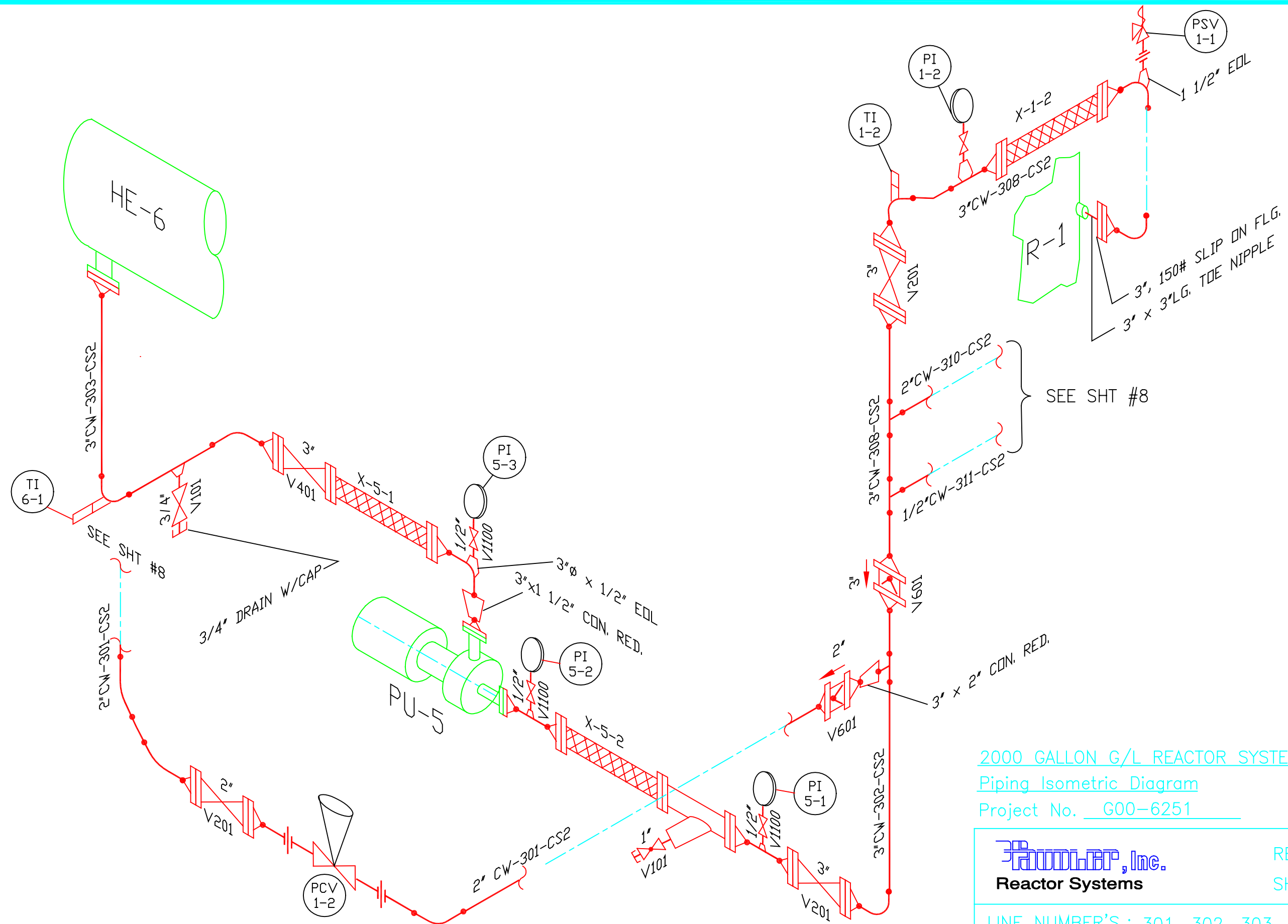
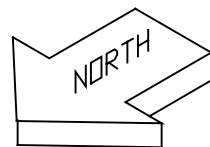
BONDING JUMPER, TYPICAL  
AT ALL FLD'D CONN.

2000 GALLON G/L REACTOR SYSTEM  
Piping Isometric Diagram  
Project No. G00-6251

**PRIMOR, Inc.**  
Reactor Systems

REV. 10/25/00  
SHT. 6 OF 9

LINE NUMBER'S : 500, & 600

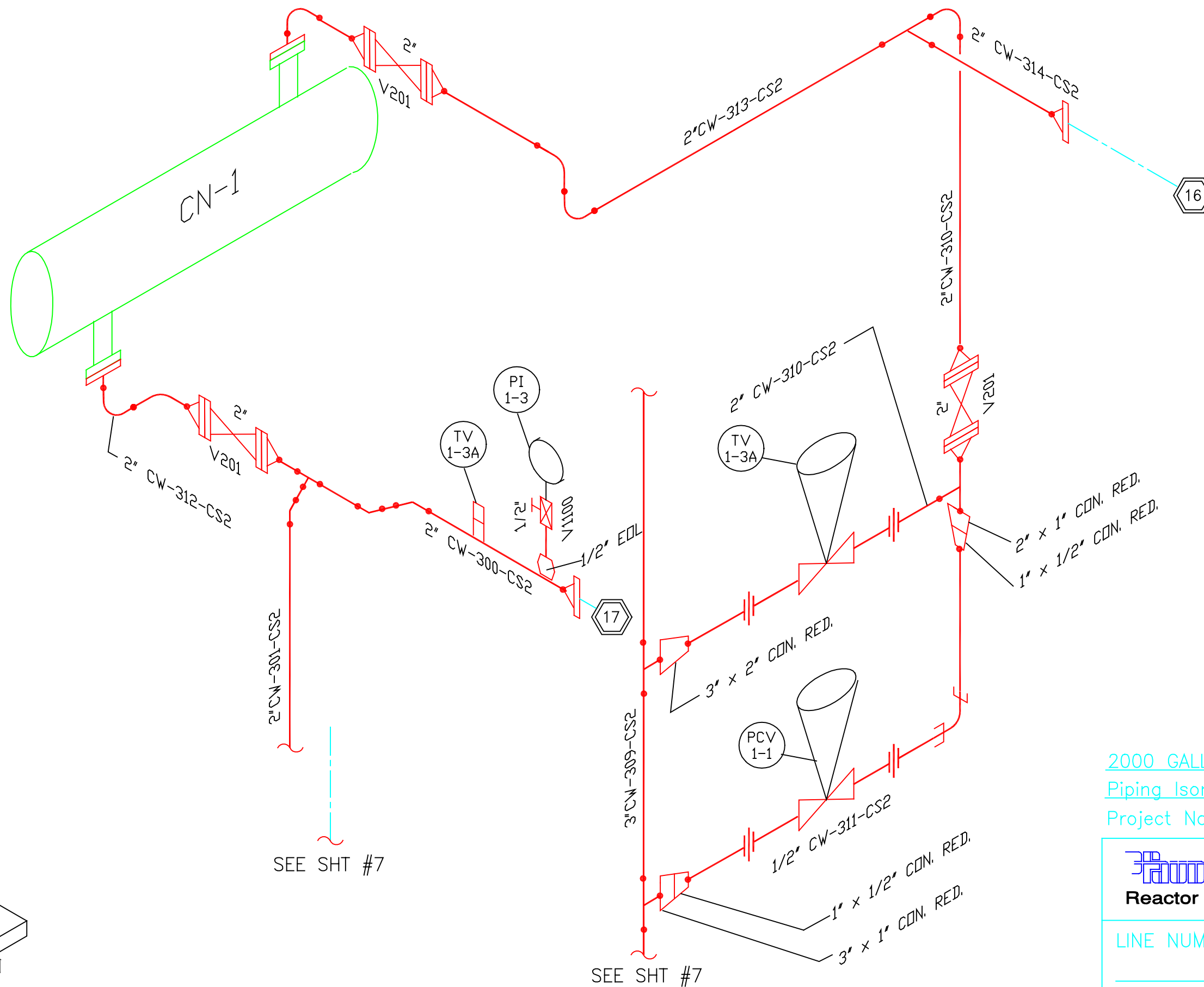
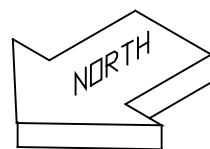


2000 GALLON G/L REACTOR SYSTEM  
Piping Isometric Diagram  
Project No. G00-6251

**Reactor Systems**

REV. \_\_\_\_\_  
SHT. 7 OF 9

LINE NUMBER'S : 301, 302, 303, 308 & 309



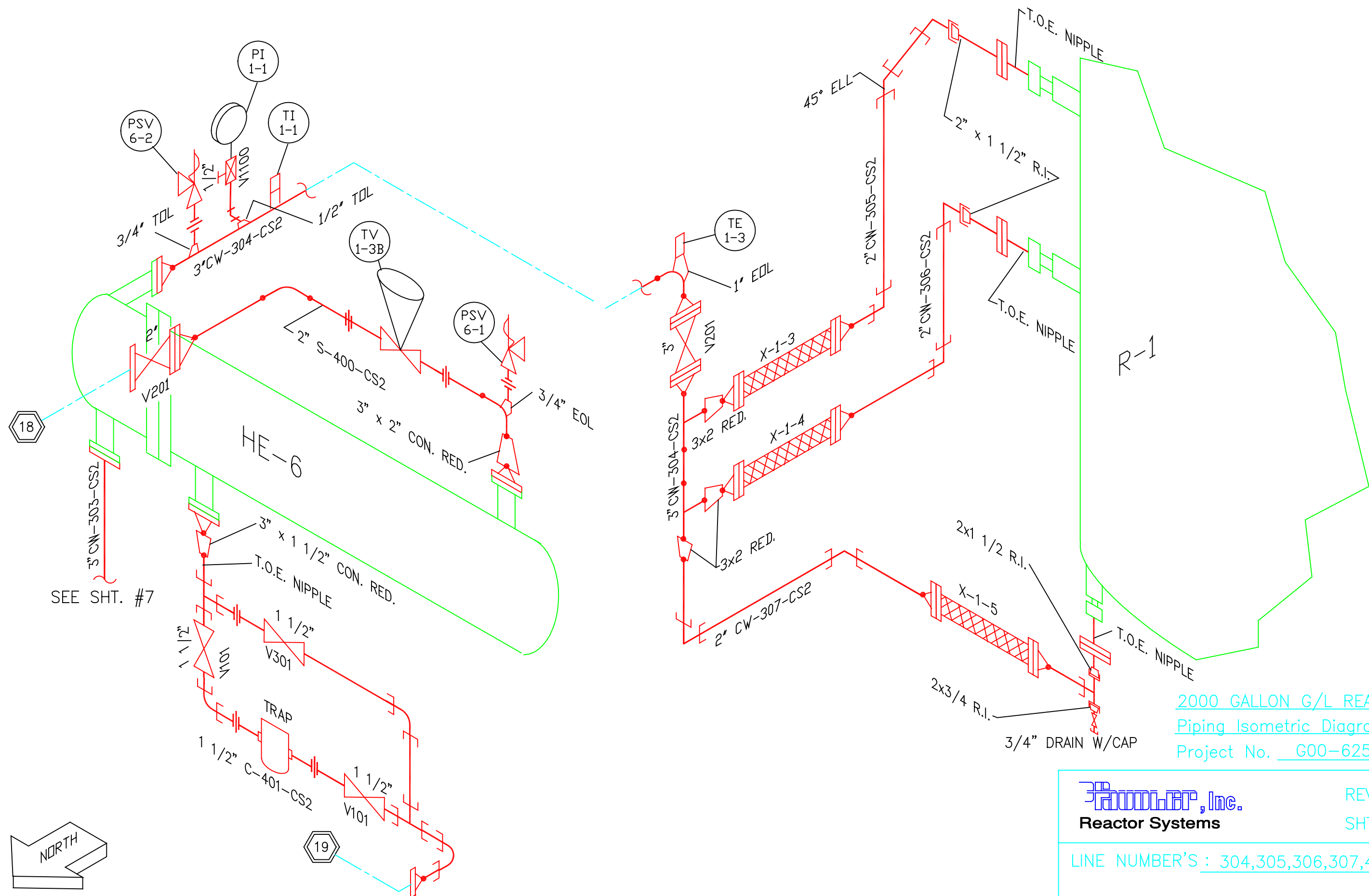
2000 GALLON G/L REACTOR SYSTEM  
Piping Isometric Diagram  
Project No. G00-6251

**Reactor Systems, Inc.**

REV. \_\_\_\_\_  
SHT. 8 OF 9

LINE NUMBER'S : 301,308,310,311,312,313, &  
314



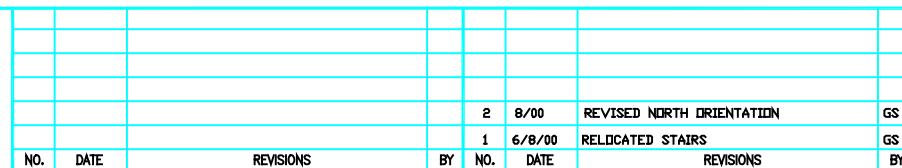
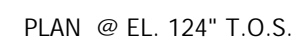
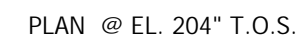


2000 GALLON G/L REACTOR SYSTEM  
Piping Isometric Diagram  
Project No. G00-6251

**Reactor Systems, Inc.**

REV. \_\_\_\_\_  
SHT. 9 OF 9

LINE NUMBER'S : 304,305,306,307,400, & 401



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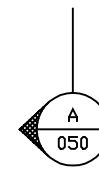
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DATE 5/31/00	APPROVED	DATE
DRAWN RL	APPROVED	DATE
WORK FROM DIMENSIONS - DO NOT SCALE		

EQUIPMENT LAYOUT - PLANS  
2000 GALLON G/L REACTOR SYSTEM  
U.S. ARMY - PICATINNY ARSENAL, NJ

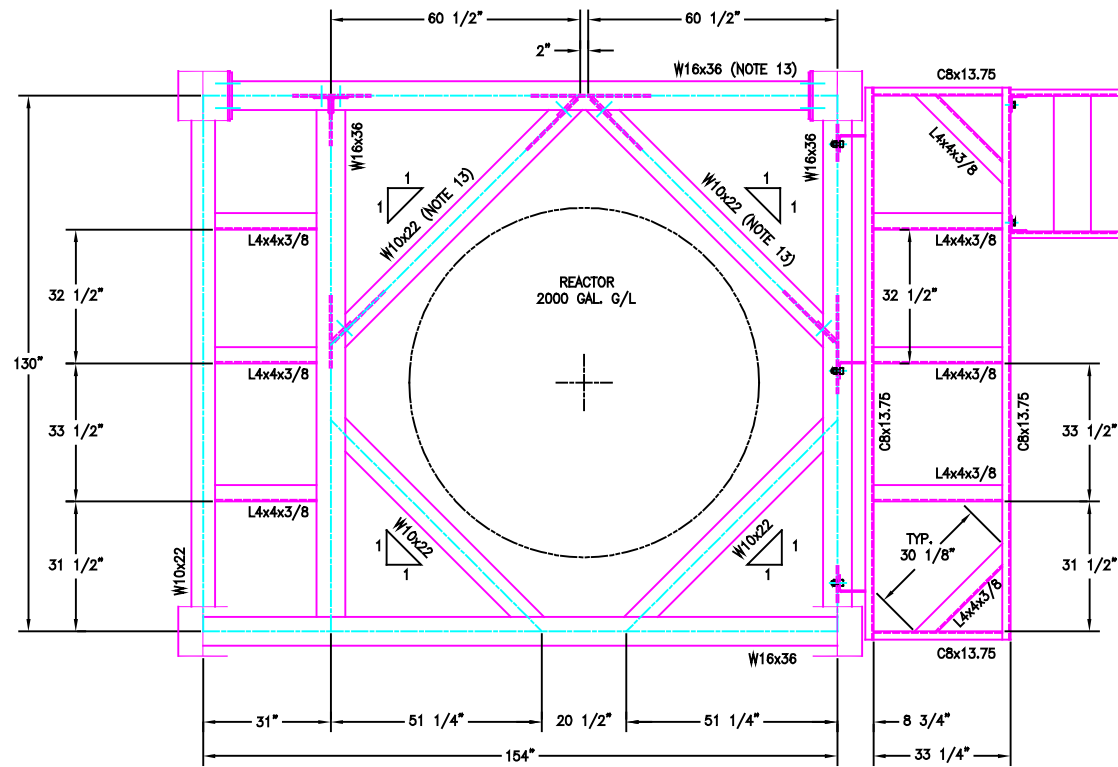


Drawing No.  
JD-00-6251-020-2

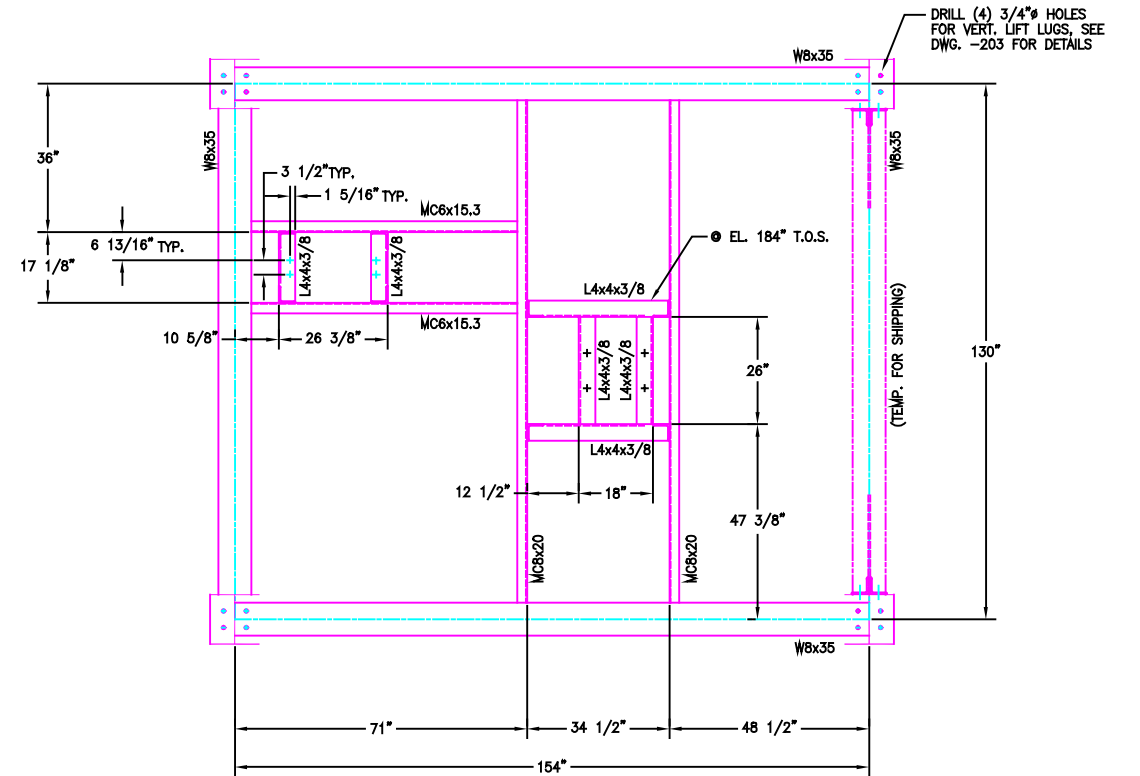




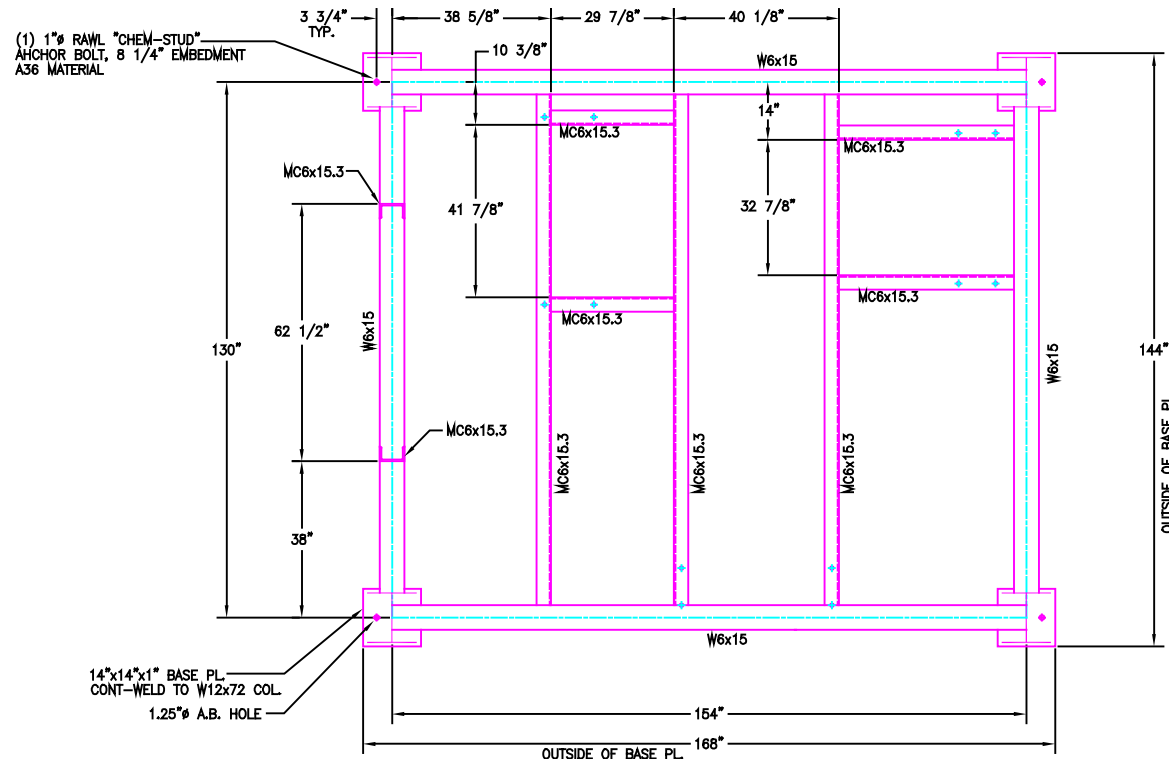
143 SQ. FT. HEAT EXCHANGER TAG: HE-6 LAYOUT  
2000 GALLON G/L REACTOR SYSTEM  
U.S. ARMY - PICATINNY ARSENAL, NJ



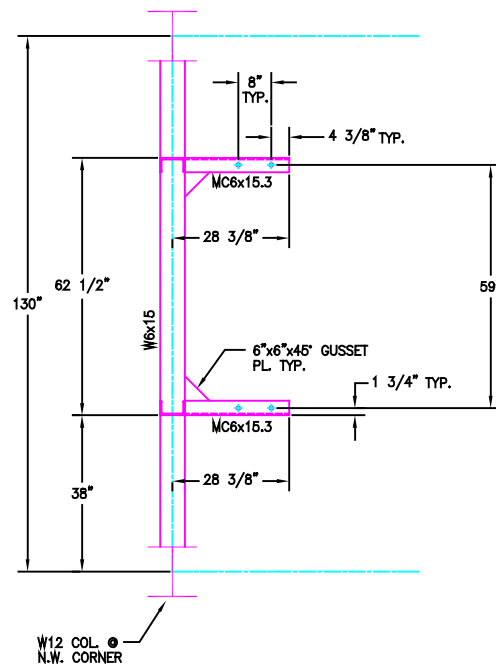
FRAMING PLAN @ EL. 124" T.O.S.



FRAMING PLAN @ EL. 204" T.O.S.



FRAMING PLAN @ EL. 8" T.O.S.



FRAMING PLAN @ EL. 64" T.O.S.

#### GENERAL NOTES:

- 1.) ALL STRUCTURAL STEEL SHALL BE DETAILED, FABRICATED AND ERECTED IN STRICT ACCORDANCE WITH THE LATEST A.I.S.C. SPECIFICATIONS. UNLESS OTHERWISE NOTED, ALL STRUCTURAL STEEL SHAPES AND PLATES SHALL CONFORM TO ASTM A36.
- 2.) UNLESS OTHERWISE NOTED, ALL BOLTED CONNECTIONS SHALL BE MADE WITH 3/4" DIAMETER HIGH-STRENGTH BOLTS CONFORMING TO ASTM A325. UNLESS OTHERWISE NOTED, ALL BOLTED CONNECTIONS SHALL BE DESIGNED AS BEARING-TYPE CONNECTIONS. ALL BOLTING SHALL BE TIGHTENED TO WITHIN 70% OF THEIR MIN. TENSILE STRENGTH.
- 3.) ALL WELDS SHALL BE MADE IN ACCORDANCE WITH THE LATEST REQUIREMENTS OF THE AWS USING E70 ELECTRODES WITH MINIMUM WELD SIZE OF 3/16" FILLET, UNLESS OTHERWISE NOTED ON DRAWINGS.
- 4.) STAIRS, LADDERS, HANDRAILS, ETC., SHALL BE DESIGNED TO MEET LATEST O.S.H.A. REGULATIONS, AND OTHER APPLICABLE CODES AND AS SHOWN ON DRAWINGS.
- 5.) ALL SHARP EDGES, BURRS, WELD SPATTER, ETC., SHALL BE REMOVED PRIOR TO APPLYING PRIMER AND FINISH COATS OF PAINT.
- 6.) THE CONTRACTOR SHALL VERIFY ALL LIFTING LUG LOCATIONS WITH STRUCTURAL ENGINEER PRIOR TO LIFTING.
- 7.) THE CONTRACTOR SHALL BE RESPONSIBLE FOR VERIFYING ALL SKID DIMENSIONS, ANY DISCREPANCIES SHALL BE BROUGHT TO THE ATTENTION OF ENGINEERED SYSTEMS PROJECT MANAGER.
- 8.) AREAS WHERE PAINT HAS BEEN REMOVED DUE TO WELDING, BURNING OR CUTTING, SHALL BE PRIMED AND REPAINTED TO MEET THICKNESS OF ORIGINAL FINISH.
- 9.) ALL BOLTING SHALL BE ASTM A325 GALVANIZED OR ZINC PLATED.
- 10.) FABRICATOR SHALL SUPPLY LIFTING LUGS AS SHOWN ON DRAWINGS.
- 11.) REACTOR LEVEL @ EL. 124" T.O.S. SHALL BE COVERED WITH 1/2" THICK STEEL DIAMOND PLATE AS SHOWN ON DRAWINGS, PLATE SHALL BE FASTENED TO STRUCTURE WITH S.S. 1/4"-20 FLAT HD. SOCKET HEAD CAP SCREW WITH MATCHING LOCK-NUTS. ATTACH PLATE TO ALL STEEL MEMBERS APPROX. 15" ON CENTERS.
- 12.) ALL SEAMS IN 1/2" DIAMOND PLATE SHALL BE BUTT-WELDED TO FORM A CONTINUOUS SURFACE, EXCEPT FOR REMOVABLE CATWALK SECTION.
- 13.) THIS MEMBER SHALL HAVE BOLTED CONNECTION TO FACILITATE REACTOR INSTALLATION.



NO.	DATE	REVISIONS	BY	NO.	DATE	REVISIONS	BY
				3	8/18/00	ADDED HE-6 & CN-1 MOUNTING HOLE DIM.	GS
				2	7/25/00	REVISED FRAMING @ EL. 204" T.O.S.	GS
				1	6/27/00	ISSUED FOR FABRICATION	GS
				0	06/12/00	ISSUED FOR CALCS.	GS

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SCALE 1=24	CHECKED	DATE
DATE 6/6/00	APPROVED	DATE
DRAWN RL	APPROVED	DATE
WORK FROM DIMENSIONS - DO NOT SCALE		



STRUCTURAL STEEL LAYOUTS - FRAMING PLANS  
2000 GALLON G/L REACTOR SYSTEM  
U.S. ARMY - PICATINNY ARSEN, NJ

Drawing No.

JD-00-6251-200-



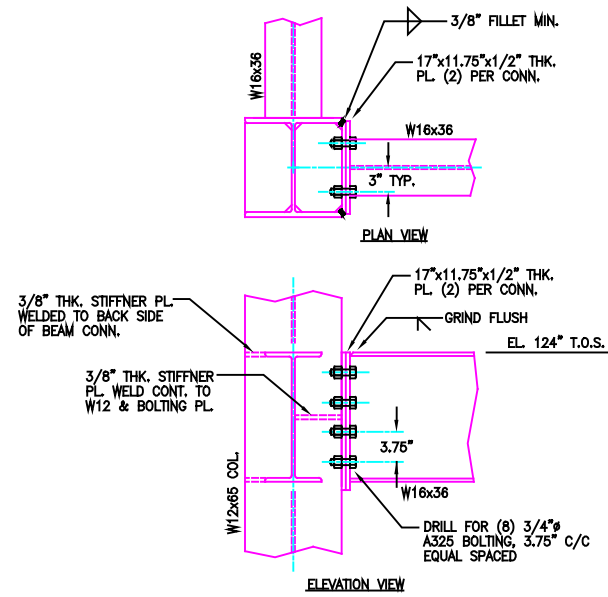
				1	06/27/00	ISSUED FOR FABRICATION	GS
				0	06/12/00	ISSUED FOR CALCS.	GS
NO.	DATE	REVISIONS	BY	NO.	DATE	REVISIONS	BY

SCALE 1=24	CHECKED	DATE
DATE 6/13/00	APPROVED	DATE
DRAWN RL	APPROVED	DATE
WORK FROM DIMENSIONS – DO NOT SCALE		

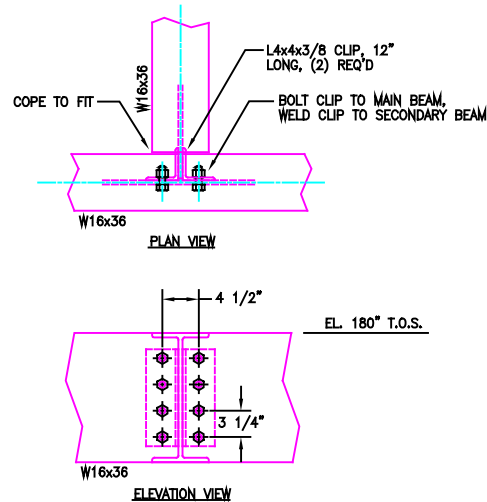
Drawing No.  
JD-00-6251-201-1



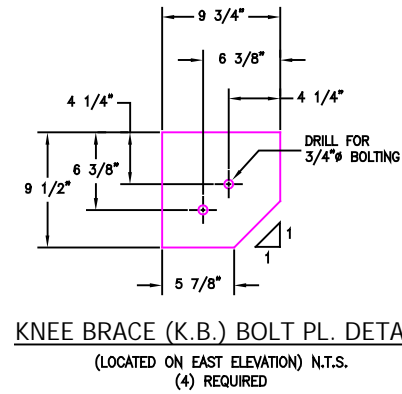




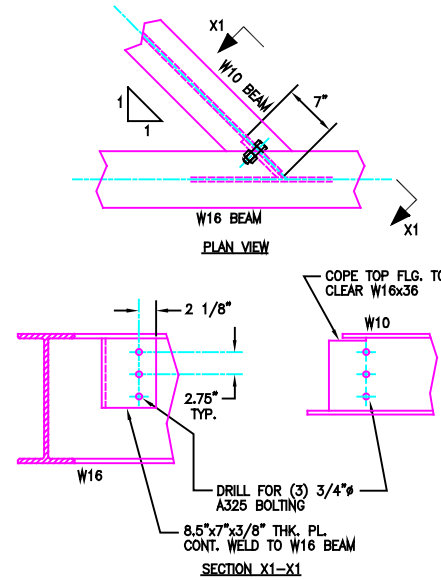
W16 BEAM to W12 COL. BOLTING DETAIL  
N.T.S. (2) REQ'D.



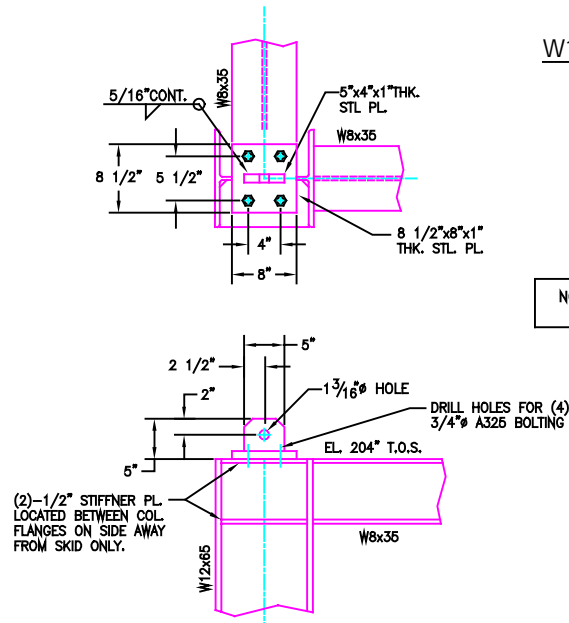
W16 to W16 BEAM BOLTING DETAIL  
N.T.S. (1) REQ'D.



KNEE BRACE (K.B.) BOLT PL. DETAIL  
(LOCATED ON EAST ELEVATION) N.T.S.  
(4) REQUIRED

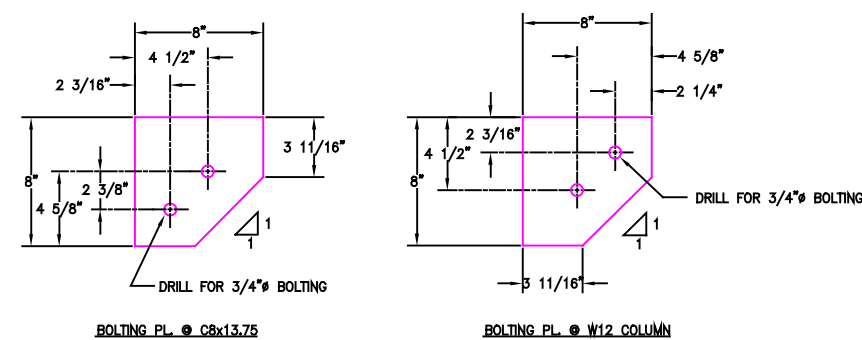


W16 to W10 @ 45° BEAM BOLTING DETAIL  
N.T.S. (4) REQ'D.

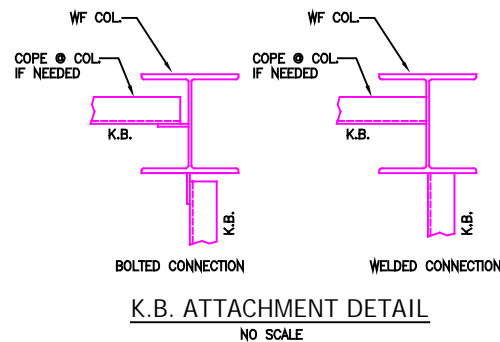


BOLTED TYPE VERT. LIFTING LUG  
FOR TOP OF W12 COLUMN  
N.T.S. (4) REQ'D.

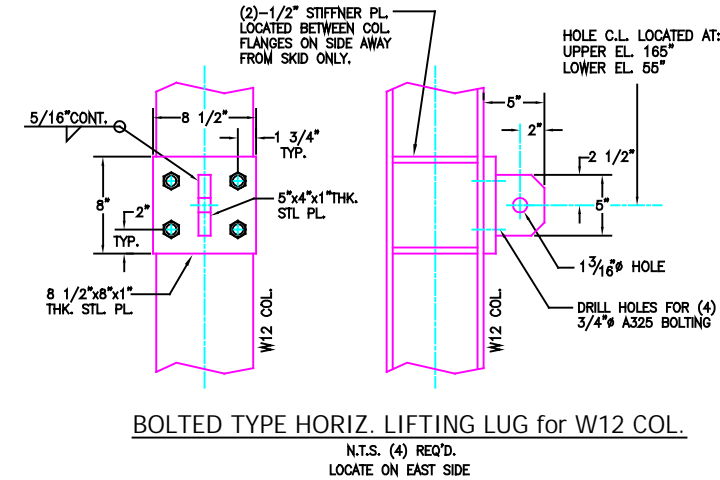
NOTE: USE MIN. LIFTING PIN/HOOK DIA. OF 1" FOR BEARING AGAINST LIFTING LUG HOLE.



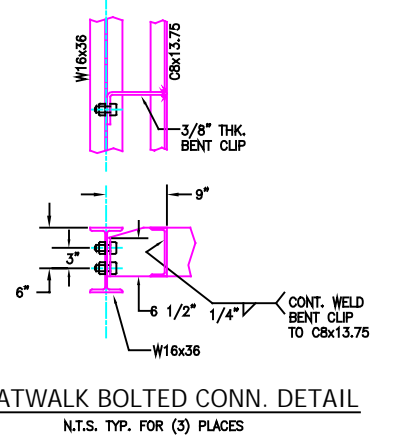
CATWALK K.B. BOLTING PLATE  
NO SCALE (2 of EACH) REQ'D



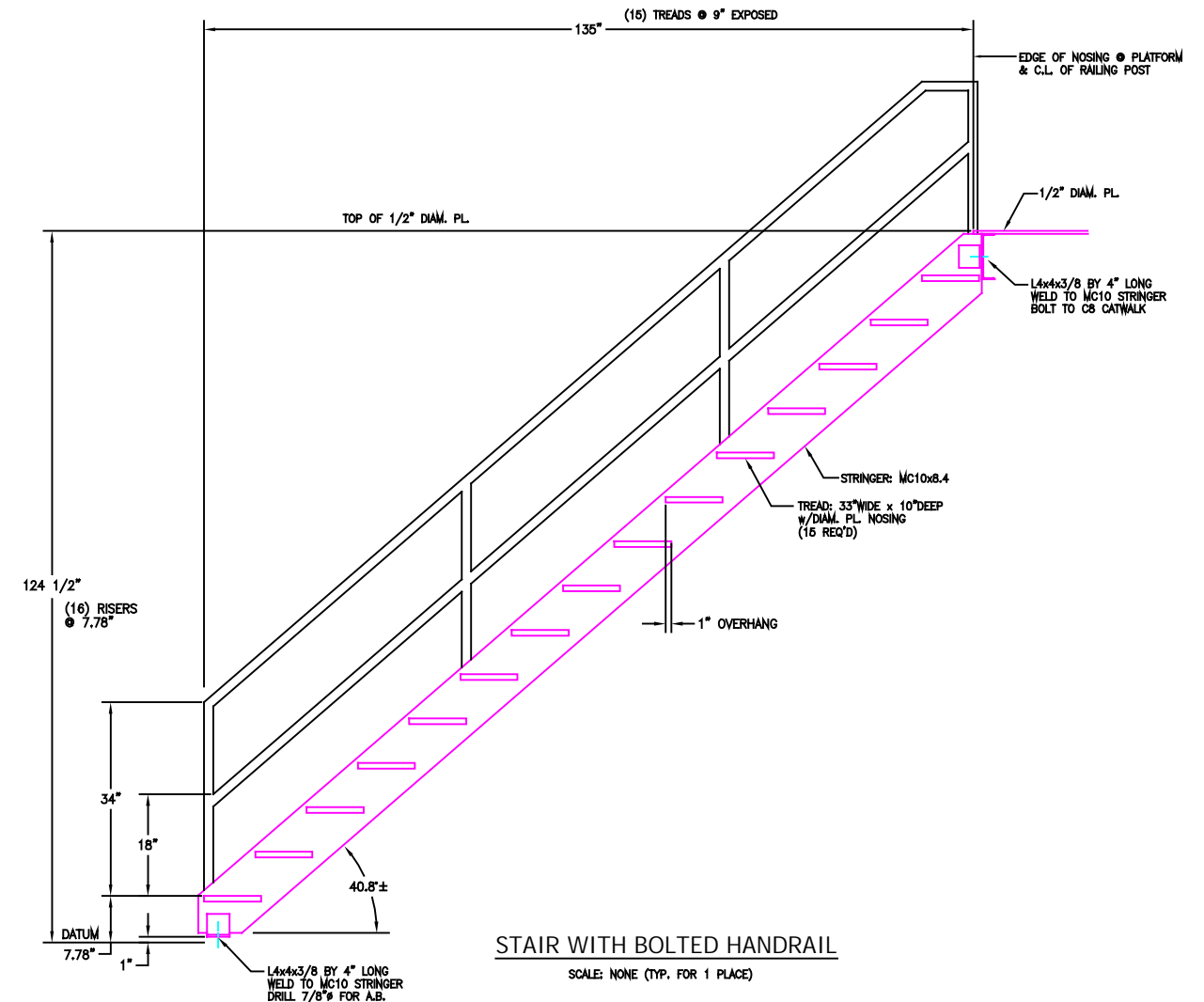
K.B. ATTACHMENT DETAIL  
NO SCALE



BOLTED TYPE HORIZ. LIFTING LUG for W12 COL.  
N.T.S. (4) REQ'D.  
LOCATE ON EAST SIDE



CATWALK BOLTED CONN. DETAIL  
N.T.S. TYP. FOR (3) PLACES




STAIR WITH BOLTED HANDRAIL  
SCALE: NONE (TYP. FOR 1 PLACE)

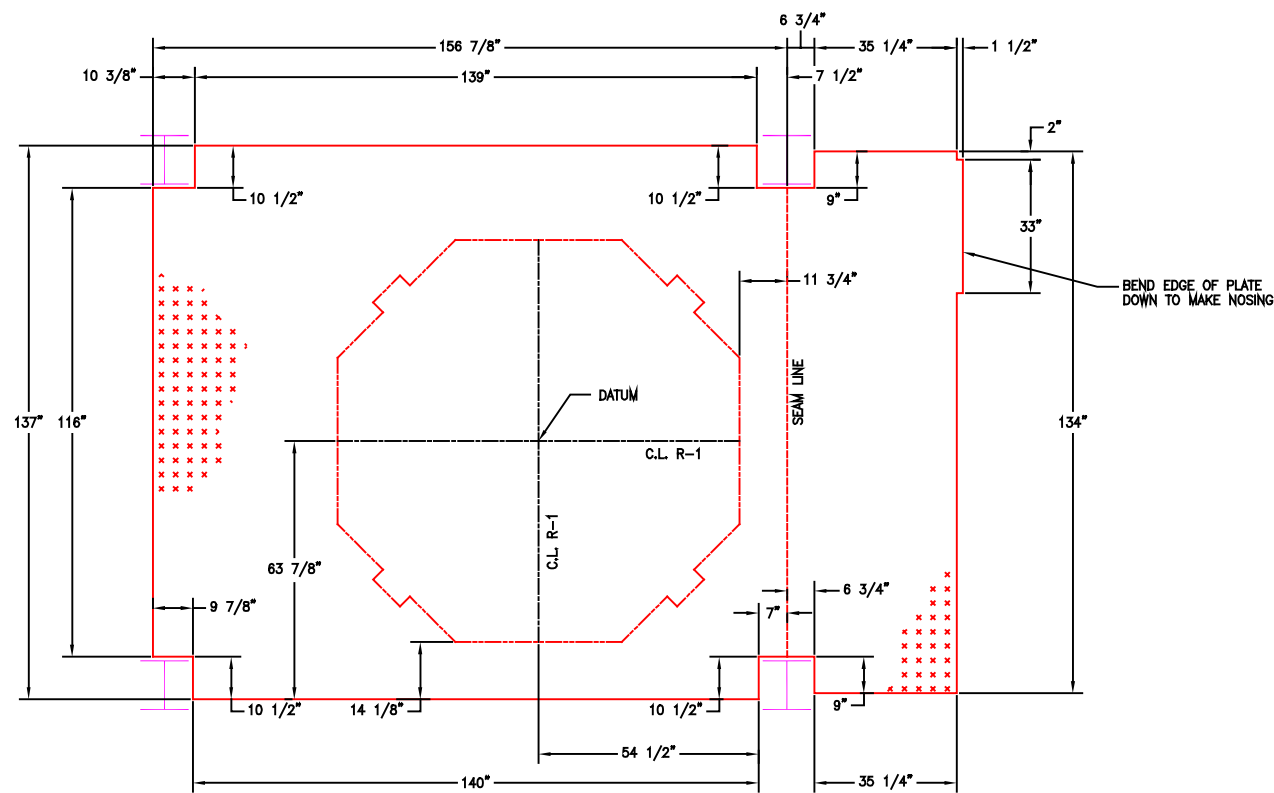
NO.	DATE	REVISIONS	BY	NO.	DATE	REVISIONS	BY
				2	7/10/00	ADDED K.B. DETAILS	GS
				1	6/27/00	ISSUED FOR FABRICATION	GS
				0	6/14/00	ISSUED FOR CALCS.	GS

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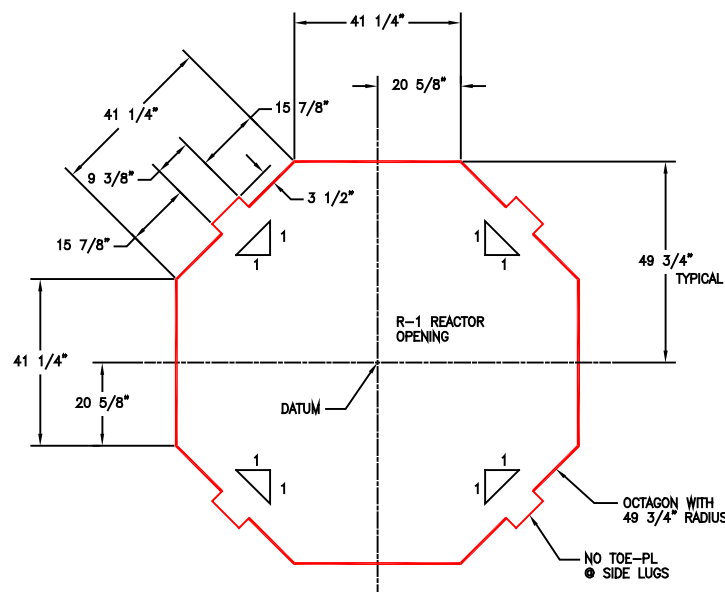
SCALE 1=24	CHECKED	DATE
DATE 6/14/00	APPROVED	DATE
DRAWN RL	APPROVED	DATE
WORK FROM DIMENSIONS - DO NOT SCALE		

STRUCTURAL STEEL LAYOUTS - MISC. DETAILS 2000 GALLON G/L REACTOR SYSTEM U.S. ARMY - PICATINNY ARSENAL, NJ		Drawing No.
		JD-00-6251-203-2

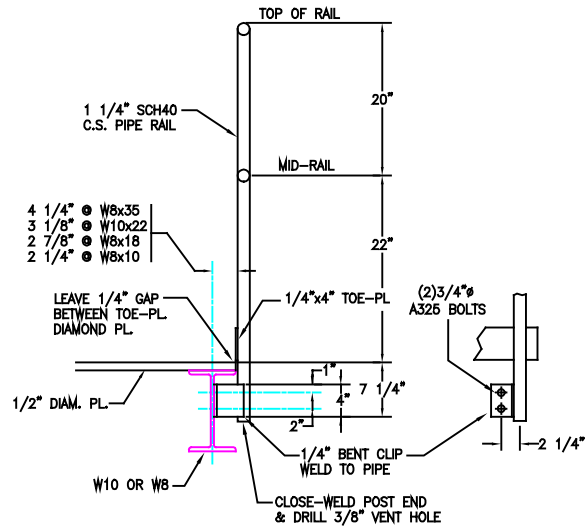




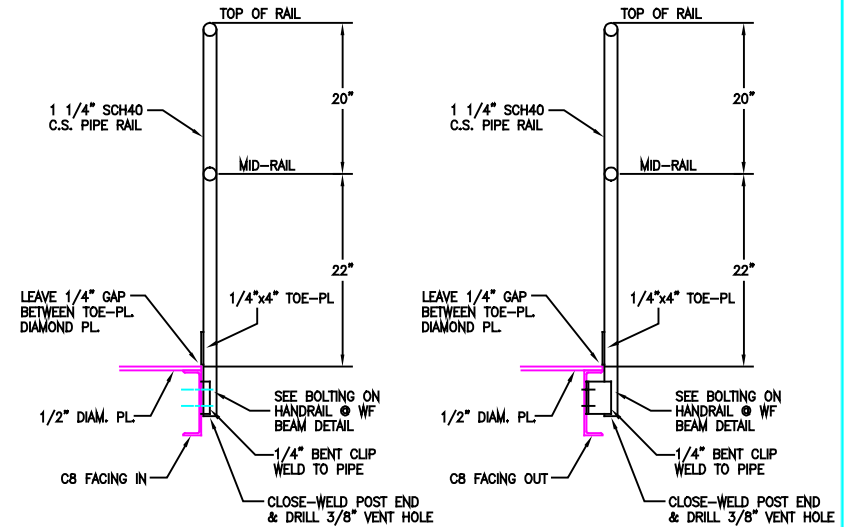
1/2" DIAMOND PLATE PLAN @ EL. 124" T.O.S.



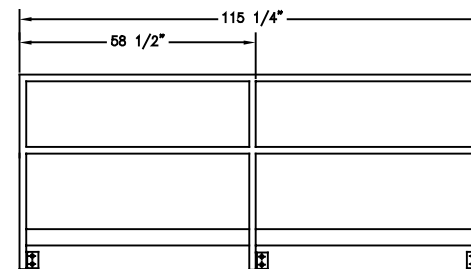
R-1 OPENING DETAIL



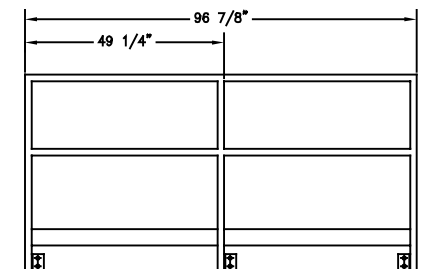
DETAIL of HAND-RAIL @ WF  
N.T.S.



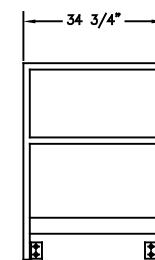
DETAIL of HAND-RAIL @ C8x13.75  
N.T.S.



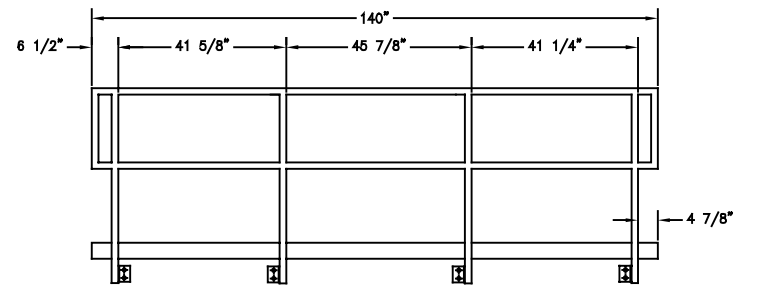
HAND-RAIL HR-1 DETAIL



HAND-RAIL HR-2 DETAIL



HAND-RAIL HR-3 & 5 DETAIL



HAND-RAIL HR-4 & 6 DETAIL

LEGEND:

- GRATING EDGED WITH TOE-PLATE, 1/4" THK. TO 4" ABOVE GRATING/INLAND PL.
- GRATING WITH NO TOE-PLATE



NO.	DATE	REVISIONS	BY	NO.	DATE	REVISIONS	BY
				1	6/27/00	ISSUED FOR FABRICATION	GS
				0	6/14/00	ISSUED FOR CALCS.	GS

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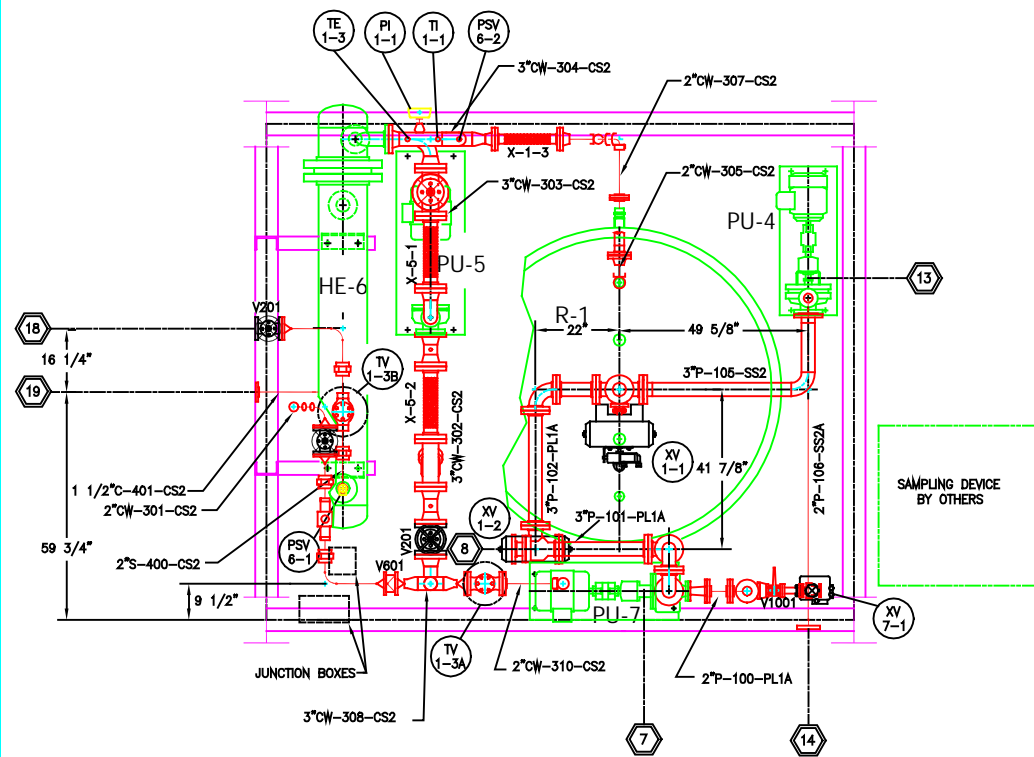
SCALE 1=24	CHECKED	DATE
DATE 6/14/00	APPROVED	DATE
DRAWN RL	APPROVED	DATE
WORK FROM DIMENSIONS - DO NOT SCALE		

STRUCTURAL STEEL LAYOUTS - GRATING, INLAND PL. & RAILING  
2000 GALLON G/L REACTOR SYSTEM  
U.S. ARMY - PICATINNY ARSENAL, NJ

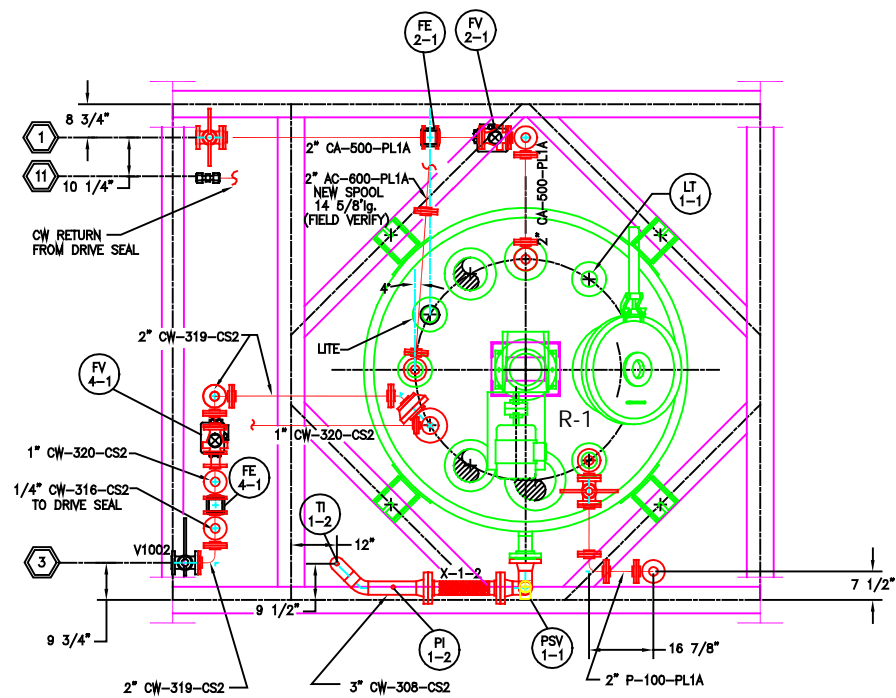


Drawing No.

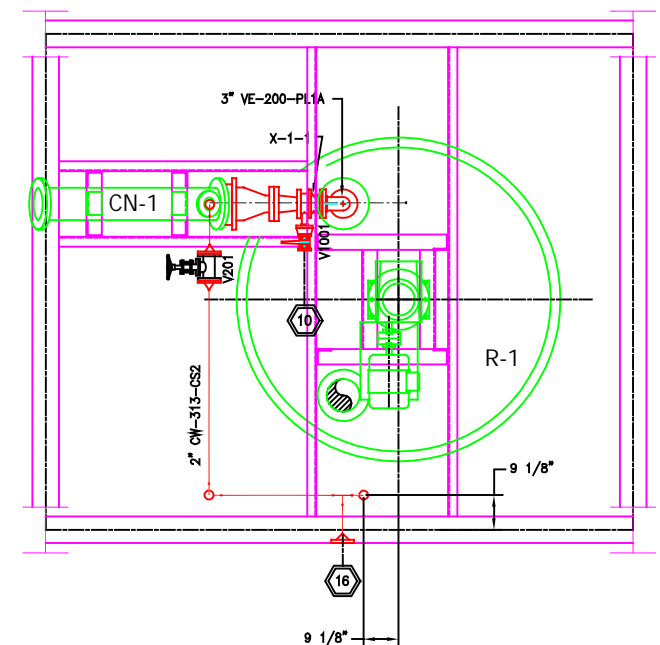
JD-00-6251-204-1



EQUIPMENT PLAN @ EL. 8" T.O.S.



EQUIPMENT PLAN @ EL. 124" T.O.S.



EQUIPMENT PLAN @ EL. 204" T.O.S.



				4	10/25/00	REVISED LINE 2" AC-600-PL1A	GS
				3	10/02/00	AS BUILT CHANGES	GS
				2	9/26/00	AS BUILT CHANGES	GS
				1	7/12/00	ISSUED FOR CONSTRUCTION	GS
				0	7/7/00	ISSUED FOR REVIEW	GS
NO.	DATE	REVISIONS	BY	NO.	DATE	REVISIONS	BY

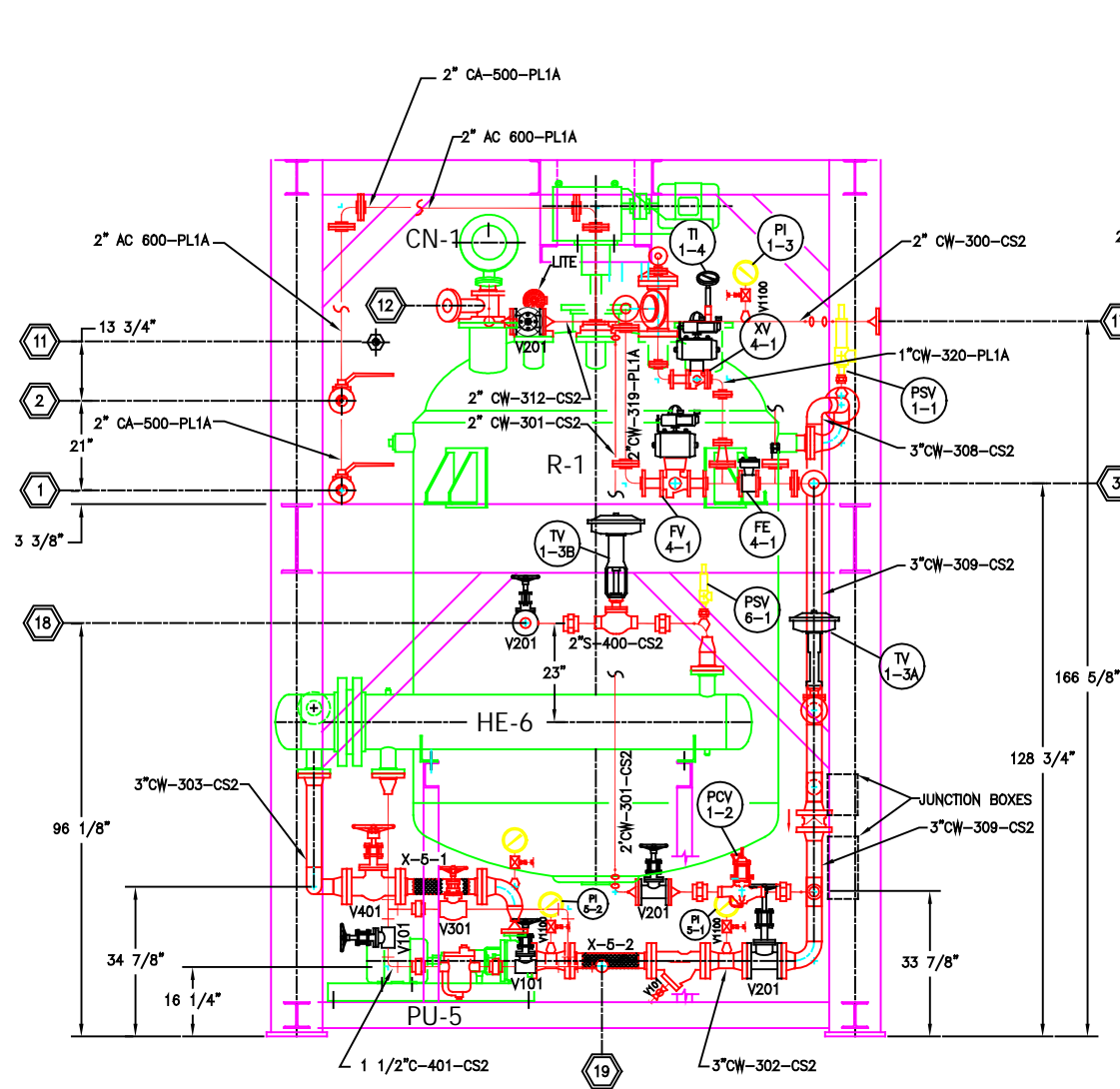
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SCALE 1=24	CHECKED	DATE
DATE 7/7/00	APPROVED	DATE
DRAWN RJD	APPROVED	DATE
WORK FROM DIMENSIONS - DO NOT SCALE		

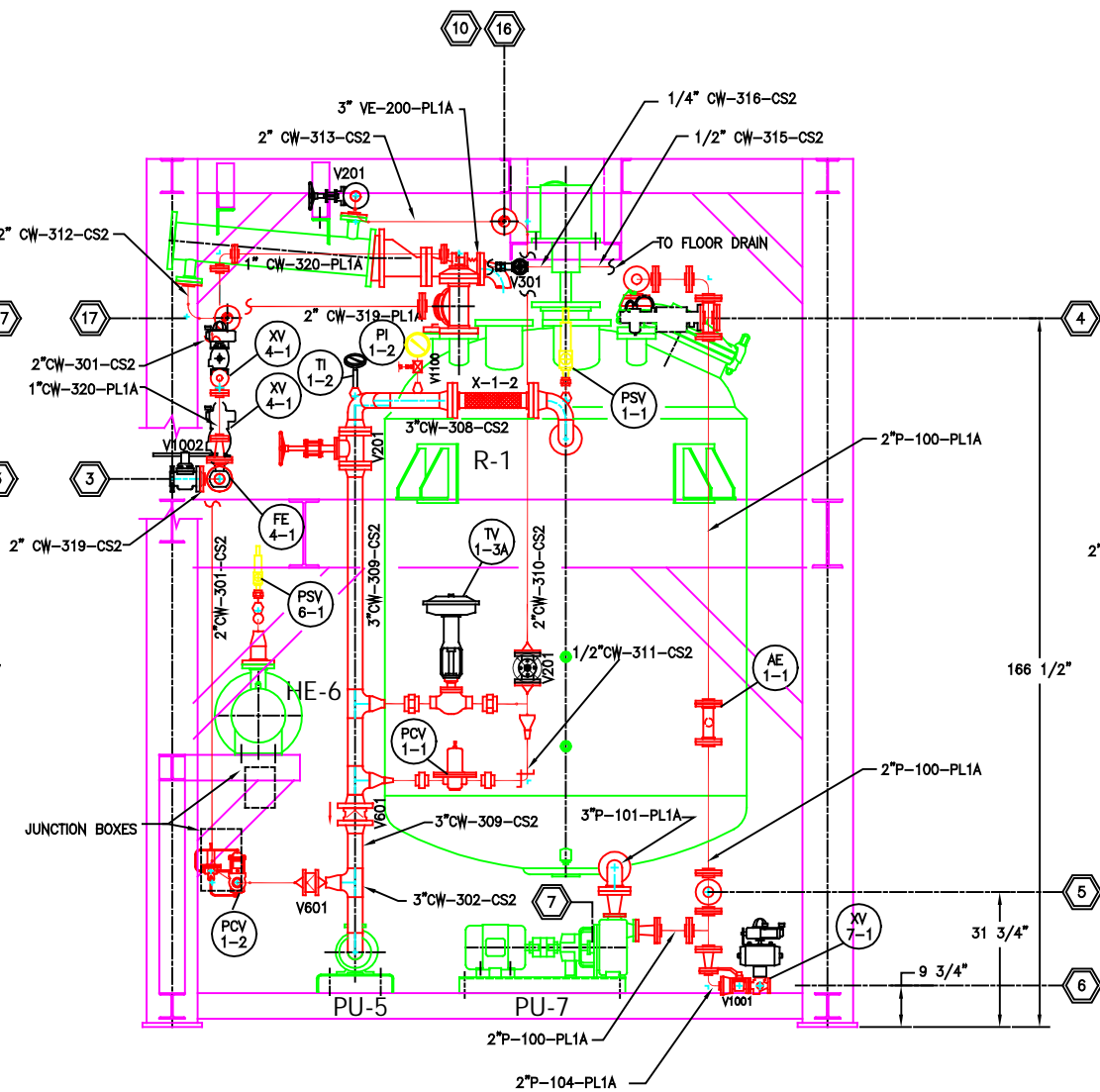
PIPING LAYOUT - PLAN VIEWS  
2000 GAL. G/L REACTOR SYSTEM  
U.S. ARMY - PICATINNY ARSENAL, N.J.



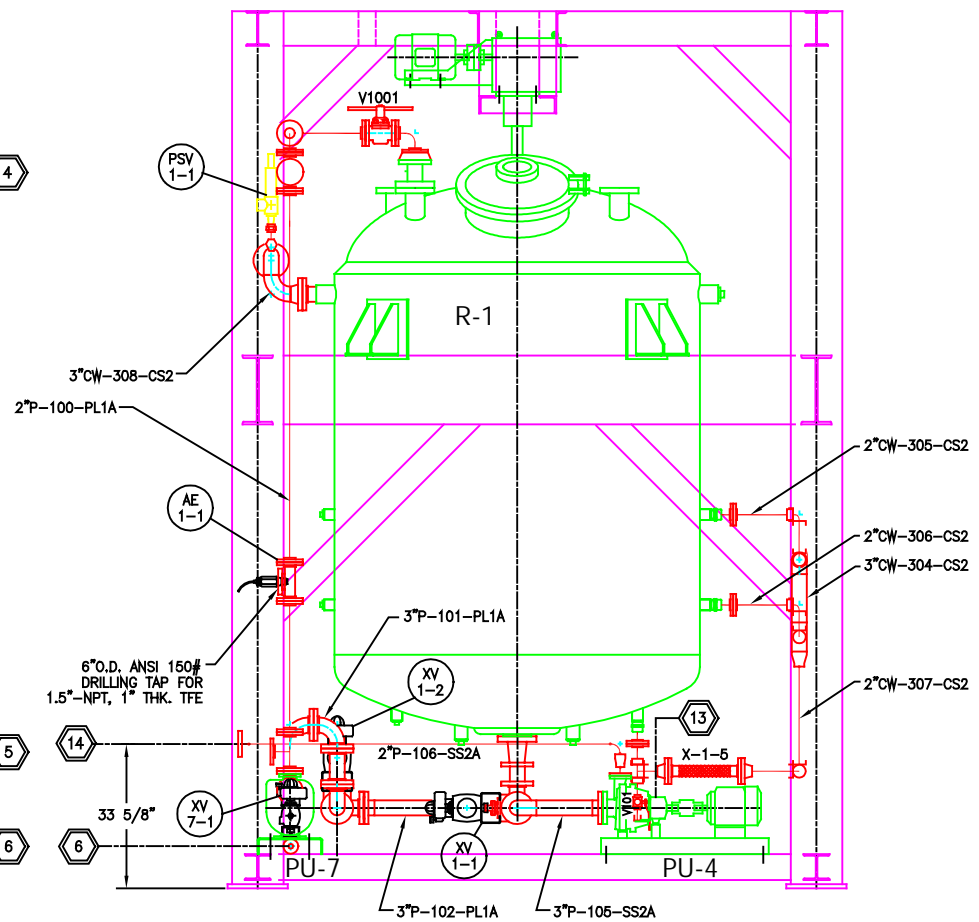
Drawing No.  
JD-00-6251-300-4



NORTH ELEVATION



WEST ELEVATION



SOUTH ELEVATION

		4	10/25/00	REVISED LINE 2" AC-600-PL1A	GS
		3	9/26/00	AS BUILT REVISIONS	GS
		2	8/4/00	REVISED PER ENG REVIEW	GS
		1	7/13/00	ISSUED FOR CONSTRUCTION	GS
		0	7/7/00	ISSUED FOR REVIEW	GS

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SCALE 1=24	CHECKED	DATE
DATE 7/7/00	APPROVED	DATE
DRAWN RL	APPROVED	DATE

PIPING LAYOUT - ELEVATIONS  
2000 GAL. G/L REACTOR SYSTEM  
U.S. ARMY - PICATINNY ARSENAL, N.J.



Drawing No.  
ID 00 6251



R-1

2000 GAL. REACTOR  
GLASSLINED CARBON STEEL  
INT. DESIGN: FV/100 PSIG @ -20°/450°F  
JKT. DESIGN: FV/100 PSIG @ -20°/450°F  
ASME CODE STAMP

AG-1

AGITATOR FOR 2000 GAL. REACTOR  
HASTELLOY C  
DUAL FLIGHT, PBT LOWER, PBT UPPER  
10 HP MOTOR (XP) CHEMINEER DRIVE

PU-4

TRANSFER PUMP  
CENTRIFUGAL TYPE  
STAINLESS STEEL  
100 GPM @ 50' TDH  
3 HP, 1800 RPM, 3PH/60Hz/460VAC, XP

PU-5

JACKET CIRCULATION PUMP  
CENTRIFUGAL TYPE  
DUCTILE IRON CONSTRUCTION  
180 GPM @ 130' TDH  
10 HP, 3600 RPM, 3P/60Hz/460VAC, XP

HE-6

STEAM HEAT EXCHANGER  
143 SQ. FT., U-TUBE TYPE  
CARBON STEEL, 304 S.S. TUBES  
FV/100 PSIG @ -20°/350°F  
ASME CODE STAMP

PU-7

RECIRCULATION PUMP  
SELF PRIMING CENTRIFUGAL  
HASTELLOY C CONSTRUCTION  
60 GPM @ 30' TDH  
3 HP, 1800 RPM, 3PH/60Hz/460VAC, XP

CN-1

VENT CONDENSER  
40 SQ. FT.  
HASTELLOY C-276 TUBESIDE  
CARBON STEEL SHELLSIDE  
FV/100 PSIG @ -20°/350°F TUBESIDE  
100 PSIG @ -20°/350°F SHELLSIDE  
ASME CODE STAMP

NOTES:  
1) VENT TO SAFE AREA.  
2) DENOTES CUSTOMER CONNECTION POINT.

			6	7/11/00	REVISED TO DATE, ADDED CUST. CONN. POINTS	EJS
			4	7/07/00	REVISED TO DATE	EJS
			3	6/22/00	REVISED TO DATE PER CUSTOMER COMMENTS	EJS
			2	6/1/00	REVISED TO DATE	EJS
			1	5/22/00	REVISED TO DATE	EJS
			0	4/26/00	ISSUED FOR REVIEW	EJS
6	9/26/00	AS BUILT REVISIONS	EJS			
NO.	DATE	REVISIONS	BY	NO.	DATE	REVISIONS

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SCALE	NONE	CHECKED		DATE	
DATE	4/26/00	APPROVED		DATE	
DRAWN	RJD	APPROVED		DATE	
WORK FROM DIMENSIONS - DO NOT SCALE					

PRELIMINARY PIPING & INSTRUMENTATION DIAGRAM  
2000 GALLON G/L REACTOR SYSTEM  
U.S. ARMY - PICATINNY ARSENAL, NJ

Drawing No.  
JD-00-6251-002-6



## INTERNATIONAL SERIES LINEARS

Power-One produces the industry's broadest selection of linear power supplies with output voltages from 5 volts through 250 volts. Rugged technology and proven design merge to create quiet, highly regulated, dependable DC power.

The International Linear series is approved to domestic and international regulatory standards, and is CE Marked to the Low Voltage Directive (LVD).

### FEATURES

- Worldwide AC Input Capabilities: 100/120/220/230/240 VAC
- $\pm 0.05\%$  Output Regulation
- Low Output Ripple
- UL, CSA, and TÜV Approvals
- Mean Time Before Failure (MTBF) in Excess of 300,000 Hours
- CE marked to Low Voltage Directive
- 100% Burn-In
- 2 Year Warranty
- Overvoltage Protection (OVP) Standard on 5V Single Outputs, Optional for other outputs under 48V



### SINGLE OUTPUT MODELS

VOLTAGE	MODEL	VOLTAGE/ CURRENT	CASE SIZE
5V	<b>HA5-1.5/OVP-A</b>	5V @ 1.5A (Note 1)	B
	<b>HB5-3/OVP-A</b>	5V @ 3A (Note 1,5)	B
	<b>HC5-6/OVP-A</b>	5V @ 6A (Note 1,5)	C
	<b>HN5-9/OVP-A</b>	5V @ 9A (Note 1,5)	N
	<b>HD5-12/OVP-A</b>	5V @ 12A (Note 1,5)	D
	<b>HE5-18/OVP-A</b>	5V @ 18A (Note 1,5)	E
	<b>F5-25/OVP-A</b>	5V @ 25A (Note 1,5,6,10)	F
	<b>G5-35/OVP-A</b>	5V @ 35A (Note 1,5,6,10)	F
12V	<b>CP197-A</b>	5V @ 50A (Note 1,5,6)	F
	<b>HA15-0.9-A</b>	12V @ 0.9A (Note 2)	B
	<b>HB12-1.7-A</b>	12V @ 1.7A (Note 5)	B
	<b>HC12-3.4-A</b>	12V @ 3.4A (Note 5)	C
	<b>HN12-5.1-A</b>	12V @ 5.1A (Note 5)	N
	<b>HD12-6.8-A</b>	12V @ 6.8A (Note 5)	D
	<b>HE12-10.2-A</b>	12V @ 10.2A (Note 5)	E
	<b>F15-15-A</b>	12V @ 16A (Note 2,5,6,10)	F
15V	<b>HA15-0.9-A</b>	15V @ 0.9A (Note 2)	B
	<b>HB15-1.5-A</b>	15V @ 1.5A (Note 5)	B
	<b>HC15-3-A</b>	15V @ 3A (Note 5)	C
	<b>HN15-4.5-A</b>	15V @ 4.5A (Note 5)	N
	<b>HD15-6-A</b>	15V @ 6A (Note 5)	D
	<b>HE15-9-A</b>	15V @ 9A (Note 5)	E
	<b>F15-15-A</b>	15V @ 15A (Note 2,5,6,10)	F

VOLTAGE	MODEL	VOLTAGE/ CURRENT	CASE SIZE
24V	<b>HA24-0.5-A</b>	24V @ 0.5A (Note 2)	B
	<b>HB24-1.2-A</b>	24V @ 1.2A (Note 5)	B
	<b>HC24-2.4-A</b>	24V @ 2.4A (Note 5)	C
	<b>HN24-3.6-A</b>	24V @ 3.6A (Note 5)	N
	<b>HD24-4.8-A</b>	24V @ 4.8A (Note 5)	D
	<b>HE24-7.2-A</b>	24V @ 7.2A (Note 5)	E
	<b>F24-12-A</b>	24V @ 12A (Note 2,5,6,10)	F
28V	<b>HA24-0.5-A</b>	28V @ 0.5A (Note 2)	B
	<b>HB28-1-A</b>	28V @ 1A (Note 5)	B
	<b>HC28-2-A</b>	28V @ 2A (Note 5)	C
	<b>HN28-3-A</b>	28V @ 3A (Note 5)	N
	<b>HD28-4-A</b>	28V @ 4A (Note 5)	D
	<b>HE28-6-A</b>	28V @ 6A (Note 5)	E
	<b>F24-12-A</b>	28V @ 10A (Note 2,5,6,10)	F
48V	<b>HB48-0.5-A</b>	48V @ 0.5A	B
	<b>HC48-1-A</b>	48V @ 1A	C
	<b>HD48-3-A</b>	48V @ 3A (Note 5)	D
	<b>HE48-4-A</b>	48V @ 4A (Note 5)	E
120V	<b>HB120-0.2-A</b>	120V @ 0.2A	B
175-210V	<b>HB200-0.12-A</b>	175-210V @ 0.12A	B
250V	<b>HB250-0.1-A</b>	250V @ 0.1A	B

- NOTES:**
- 1) Overvoltage protection provided. Set at 6.2V  $\pm 0.4$ V.
  - 2) Adjustable outputs: F15-15-A: 12-15V, F24-12-A: 24-28V, HA15-0.9-A: 12-15V, HA24-0.5-A: 24-28V.
  - 3) Nonadjustable 3 terminal regulator.
  - 4) Isolated outputs, can be referenced as positive (+) or negative (-).
  - 5) Remote sense provided.

- 6) With output inhibit and parallel operation master/slave capability.
- 7) With output inhibit.
- 8) Adjustable 3 terminal regulator.
- 9) Can be made into an isolated output by removing jumper W1.
- 10) Model requires 100 LFM forced air cooling above 75% of rated output power.

## INTERNATIONAL SERIES LINEARS

### DUAL OUTPUT MODELS

VOLTAGE	MODEL	OUTPUT # 1	OUTPUT # 2	CASE SIZE
±5V	<b>HAA5-1.5/OVP-A</b>	+5V @ 1.5A (Note 1)	-5V @ 1.5A (Note 1)	AA
	<b>HB85-3/OVP-A</b>	+5V @ 3A (Note 1)	-5V @ 3A (Note 1)	BB
	<b>HCC5-6/OVP-A</b>	5V @ 6A (Note 1,4,5)	-5V @ 6A (Note 1,4,5)	CC
± 12V	<b>HAD12-0.4-A</b>	+12V @ 0.4A (Note 3)	-12V @ 0.4A (Note 3)	B
± 15V	<b>HAD15-0.4-A</b>	+15V @ 0.4A (Note 3)	-15V @ 0.4A (Note 3)	B
±12V to ±15V	<b>HAA15-0.8-A</b>	+12V @ 1A or +15V @ 0.8A (Note 5)	-12V @ 1A or -15V @ 0.8 or -5V @ 0.4A (Note 5)	AA
	<b>HB815-1.5-A</b>	+12V @ 1.7A or +15V @ 1.5A (Note 5)	-12V @ 1.7A or -15V @ 1.5A or -5V @ 0.7A (Note 5)	BB
	<b>HCC15-3-A</b>	+12V @ 3.4A or +15V @ 3A (Note 5)	-12V @ 3.4A or -15V @ 3A (Note 5)	CC
	<b>HDD15-5-A</b>	+12V or 15V @ 5A (Note 5)	(-)12V or 15V @ 5A	E
±18V ±24V	<b>HAA24-0.6-A</b>	+18-20V @ 0.4A or +24V @ 0.6A	(-)18-20V @ 0.4A or -24V @ 0.6A	AA
	<b>HB824-1.2-A</b>	+18-20V @ 0.9A or +24V @ 1.2A	(-)18-20V @ 0.9A or -24V @ 1.2A	BB
	<b>HCC24-2.4-A</b>	+18-20V @ 1.8A +24V @ 2.4A (Note 5)	(-)18-20V @ 1.8A -24V @ 2.4A (Note 5)	CC
5V and 12V-15V	<b>HAA512-A</b>	5V @ 2A (Note 1,4,5)	12-15V @ 0.5A (Note 4)	AA
	<b>HB8512-A</b>	5V @ 3A (Note 1,4,5)	12-15V @ 1.25A (Note 4,5)	BB
	<b>HCC512-A</b>	5V @ 6A (Note 1,4,5)	12-15V @ 2.5A (Note 4,5)	CC

### TRIPLE OUTPUT MODELS

MODEL	OUTPUT # 1	OUTPUT # 2	OUTPUT # 3	CASE SIZE
<b>HTAA-16W-A</b>	5V @ 2A (Note 1,4)	+12 to 15V @ 0.4A	(-)12 to 15V @ 0.4A or -5V @ 0.4A	AA
<b>HBAA-40W-A</b>	5V @ 3A (Note 1,4,5)	+12V @ 1A or +15V @ 0.8A (Note 5)	-12V @ 1A or -15V @ 0.8A or -5V @ 0.4A (Note 5)	BAA
<b>HCAA-60W-A</b>	+5V @ 6A (Note 1,5)	+12 to 15V @ 1A	(-)12 to 15V @ 1A or -5V @ 0.4A	D
<b>HCBB-75W-A</b>	5V @ 6A (Note 1,4,5)	+12V @ 1.7A or +15V @ 1.5A (Note 5)	-12V @ 1.7A or -15V @ 1.5A or -5V @ 0.7A (Note 5)	CBB
<b>CP131-A</b>	5V @ 8A (Note 1,4,5)	+12V @ 1.7A or +15V @ 1.5A (Note 5)	-12V @ 1.7A or -15V @ 1.5A or -5V @ 0.7A (Note 5)	131
<b>HDBB-105W-A</b>	5V @ 12A (Note 1,4,5)	+12V @ 1.5A or +15V @ 1.5A (Note 5)	-12V @ 1.7A or -15V @ 1.5A or -5V @ 0.7A (Note 5)	DBB
<b>HDCC-150W-A</b>	5V @ 12A (Note 1,4,5)	+12V @ 3.4A or +15V @ 3A (Note 5)	-12V @ 3.4A or -15V @ 3A	DCC

NOTES: 1) Overvoltage protection provided. Set at 6.2V ±0.4V.

2) Adjustable outputs: F15-15-A: 12-15V, F24-12-A: 24-28V,  
HA15-0.9-A: 12-15V, HA24-0.5-A: 24-28V.

3) Nonadjustable 3 terminal regulator.

4) Isolated outputs, can be referenced as positive (+) or negative (-).

5) Remote sense provided.

6) With output inhibit and parallel operation master/slave capability.

7) With output inhibit.

8) Adjustable 3 terminal regulator

9) Can be made into an isolated output by removing jumper W1.

## INTERNATIONAL SERIES LINEARS

### HIGH PEAK MODELS

MODEL	OUTPUT # 1	OUTPUT # 2	OUTPUT # 3	OUTPUT # 4	CASE SIZE
CP323-A*	+5V @ 2A (Note 1)	+12 @ 4A (Note 7)			N
CP205-A*	+5V @ 1A (Note 1)	-5V @ 0.5A (Note 1)	24V @ 1.5A/ or 1.7A @ PK (Note 4)		BAA
CP162-A*	+5V @ 3A (Note 1,5)	-5V @ 0.6A (Note 1)	24V @ 5A/ or 6APK (Note 4,5)		CP131
CP510-A*	+5V @ 6A (Note 1)	+12V @ 2.5A/ 7.5APK			CP510-A
CP379-A*	+5V @ 6A (Note 1,5,9)	-5V @ 1.2A or -12V @ 1.2A (Note 8)	24V @ 3.5A/ 8APK (Note 4,5)		CP131
CP498-A*	+5V @ 6A (Note 1,5)	+12V @ 5A/10APK (Note 5)	12V @ 0.5A or 5V @ 0.25A (Note 4,8)		CP131
CP503-A*	+5V @ 6A (Note 1)	+12V @ 1A	-12V @ 1A or -5V @ 0.5A	+24V @ 2.4A/ 4APK (Note 9)	CP131

NOTES: 1) Overvoltage protection provided. Set at 6.2V  $\pm$  0.4V.

2) Adjustable outputs: F15-15-A: 12-15V, F24-12-A: 24-28V,  
HA15-0.9-A: 12-15V, HA24-0.5-A: 24-28V.

3) Nonadjustable 3 terminal regulator.

4) Isolated outputs, can be referenced as positive (+) or negative (-).

\* Non-stocked standards

5) Remote sense provided.

6) With output inhibit and parallel operation master/slave capability.

7) With output inhibit.

8) Adjustable 3 terminal regulator

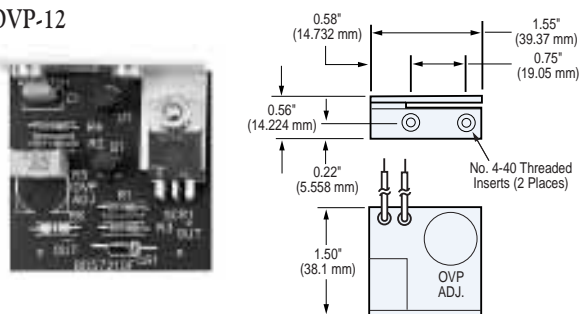
9) Can be made into an isolated output by removing jumper W1.

### OVERVOLTAGE PROTECTION OPTIONS

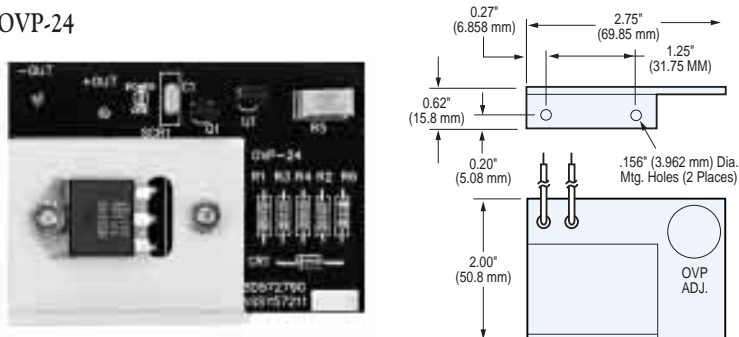
#### OVERVOLTAGE PROTECTION OPTIONS

These optional overvoltage protection modules are offered for use with Power-One's International Series Linear power supplies. Each is user adjustable from 6.4V to 34V.

##### OVP-12



##### OVP-24



#### OVP SELECTION GUIDE

MODEL	CASE SIZE	OVP MODULES REQUIRED
SINGLE OUTPUT	B,C,N,D	(1) OVP-12
	E,F	(1) OVP-24
DUAL OUTPUT	AA,B,BB,CC	(1) OVP-12 protects both outputs
	E	(1) OVP-24 protects both outputs
TRIPLE OUTPUT	AA,BAA,D CBB, 131 DBB,DCC	(1) OVP-12 protects both 12V through 15V outputs
PEAK CURRENT MODELS	N,BAA,CBB 131, CP340-A CP510-A	(1) OVP-12 protects any output not provided with built-in OVP

NOTE: Outputs with factory built-in OVP are indicated in the Voltage/Current Rating Chart for each model. OVP is not available for 48V through 250V models.

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24 Hours/Day—7 Days/Week



## INTERNATIONAL SERIES LINEARS

### INPUT SPECIFICATIONS

PARAMETER	CONDITIONS/DESCRIPTION	MIN	NOM	MAX	UNITS
Input Voltage - AC (Note 1, 2)	Jumper selectable, shipped factory configured for 120VAC operation. All models must be correctly fused for proper operation.	100 VAC Tap	87	100	110
		120 VAC Tap	104	120	132
		220 VAC Tap	191	220	242
		240 VAC Tap	209	240	264
Input Frequency	AC input.	47		63	Hz
Line Regulation	Output voltage change for a 10% line change: F case models. HAD12, HAD15. Outputs with adjustable three terminal regulators. All other models.	-0.01		+0.01	
		-1.0		+1.0	%
		-0.5		+0.5	
		-0.05		+0.05	

**NOTES:** 1) Derate output current 10% for 50Hz operation.  
2) Input voltage tolerance for 230VAC operation is +15%, -10%.

### OUTPUT SPECIFICATIONS

PARAMETER	CONDITIONS/DESCRIPTION	MIN	NOM	MAX	UNITS
Output Adjustment	Minimum output adjustment range (Note 1).	-5		+5	%
Efficiency	5 volt outputs.		45		
	12 volt and 15 volt outputs.		55		%
	24 volt and higher outputs.		60		
Ripple and Noise (Note 2)	F case models.			3.0	mVPK-PK
	5 volt, 12 volt, and 15 volt models.			5.0	mVPK-PK
	All three terminal regulator outputs.			0.2	%PK-PK
	24 volt through 250 volt models.	3.0mVPK-PK plus 0.02% of output voltage, max			
Load Regulation	Output change for a 50% load change: F case models. HAD12, HAD15. Outputs with adjustable three terminal regulators. All other models.	-0.02		+0.02	
		-1		+1	%
		-0.5		+0.5	
		-0.05		+0.05	
Transient Response	Recovery time, to within 1% of initial set point due to a 50% load change.			50	μS

**NOTES:** 1) Voltages from 3 terminal regulator outputs are not adjustable on HAD12 and HAD15.  
2) Full load, 20MHz bandwidth.

### SAFETY, REGULATORY, AND EMI SPECIFICATIONS

PARAMETER	CONDITIONS/DESCRIPTION	MIN	NOM	MAX	UNITS
Agency Approvals	UL1950.				
	CSA 1402 or CSA 22.2 No. 234/950.			Approved	
	EN60950 (TÜV).				
Dielectric Withstand Voltage	Input to output.	3750			V <sub>RMS</sub>
	Input to ground.	3750			
Electromagnetic Interference	FCC CFR title 47 Part 15 Sub-Part J - conducted. EN55022 / CISPR 22 conducted. EN55022 / CISPR 22 radiated.	Compatible with system compliance to Level B.			
Leakage Current	Per EN60950 (264VAC)		23	50	μA

### INTERFACE SIGNALS AND INTERNAL PROTECTION

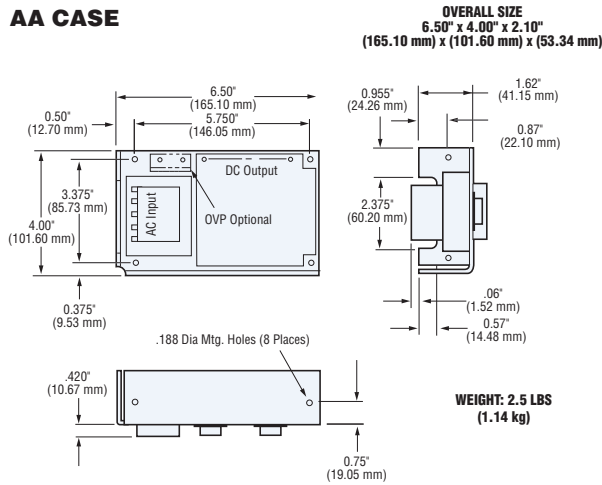
PARAMETER	CONDITIONS/DESCRIPTION	MIN	NOM	MAX	UNITS
Overvoltage Protection	Provided on 5 volt output units where indicated. Other outputs may use optional overvoltage protectors OVP-12 and OVP-24.	5.8		6.6	V
Remote Sense	Total voltage compensation for cable losses with respect to the main output. Provided on models where indicated.			250	mV
Overcurrent/Short Circuit Protection	Automatic current limit/foldback. Rated as a percentage of output power.	115	120	140	%
Master/Slave Operation	For parallel operation of up to 6 units. Master/slave pin provided on F case models only. Contact factory for application notes.				

### ENVIRONMENTAL SPECIFICATIONS

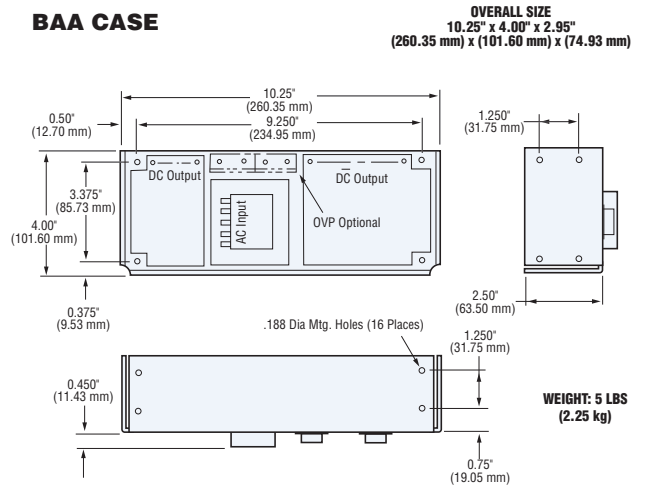
PARAMETER	CONDITIONS/DESCRIPTION	MIN	NOM	MAX	UNITS
Operating Temperature	Derate output power linearly above 50°C by 3% per °C.			50	°C
	At 100% load At 40% load			70	°C
Storage Temperature		-40		85	°C
Temperature Coefficient	0°C to 50°C (after 15 minute warm-up). 24 hours after warm-up.		0.1	0.3	%/°C
		-0.3		+0.3	%
Shock	Operating.			20	GPK
Vibration	Random vibration from 10Hz to 2kHz, 3 axis.			6.15	GRMS

## INTERNATIONAL SERIES LINEARS

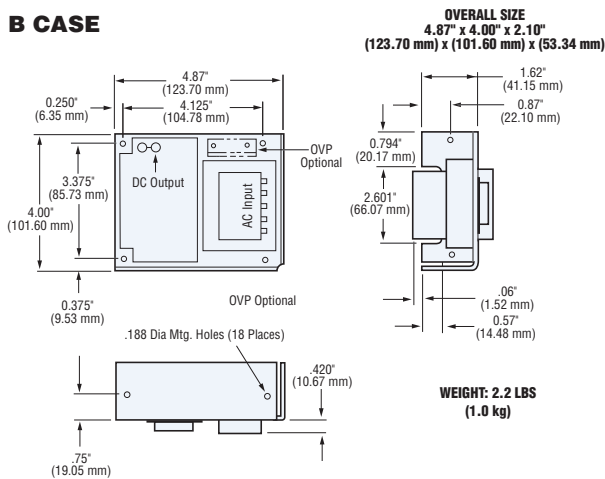
### AA CASE



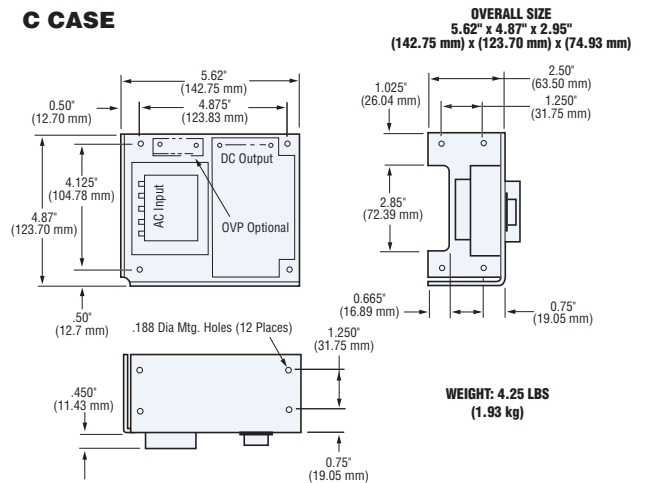
### BAA CASE



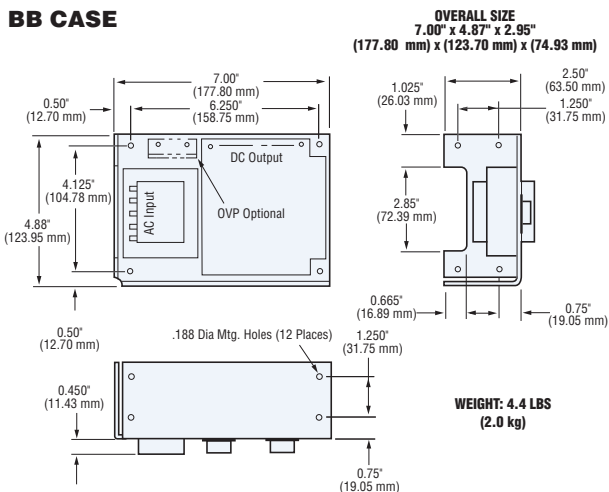
### B CASE



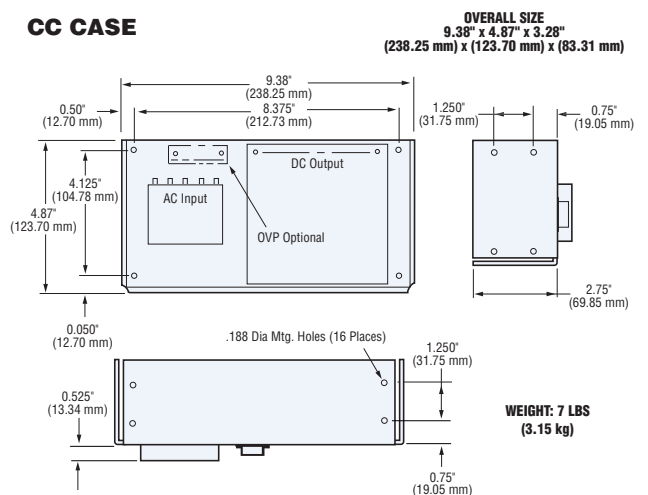
### C CASE



### BB CASE



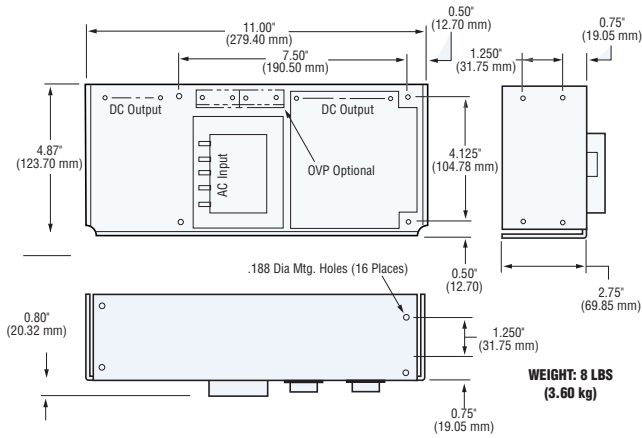
### CC CASE



## INTERNATIONAL SERIES LINEARS

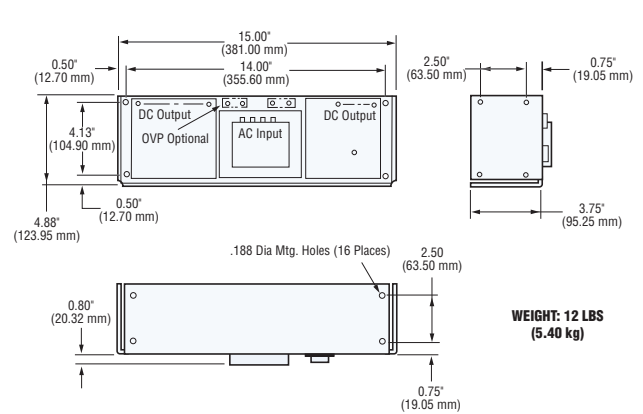
### CBB CASE

**OVERALL SIZE**  
11.00" x 4.87" x 3.28"  
(279.40 mm) x (123.70 mm) x (83.91 mm)



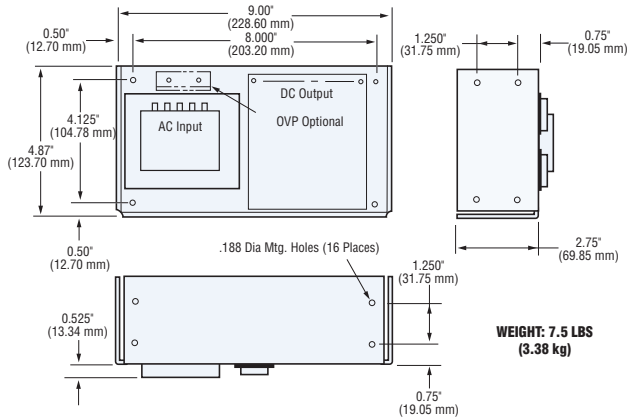
### DCC CASE

**OVERALL SIZE**  
15.00" x 4.88" x 4.55"  
(381.00 mm) x (123.95 mm) x (115.57 mm)



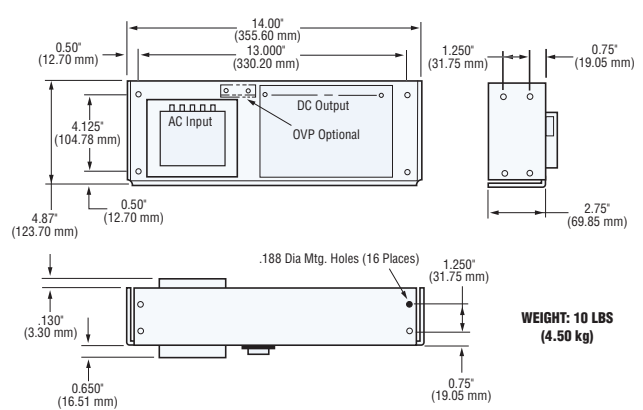
### D CASE

**OVERALL SIZE**  
9.00" x 4.87" x 3.28"  
(228.60 mm) x (123.70 mm) x (83.31 mm)



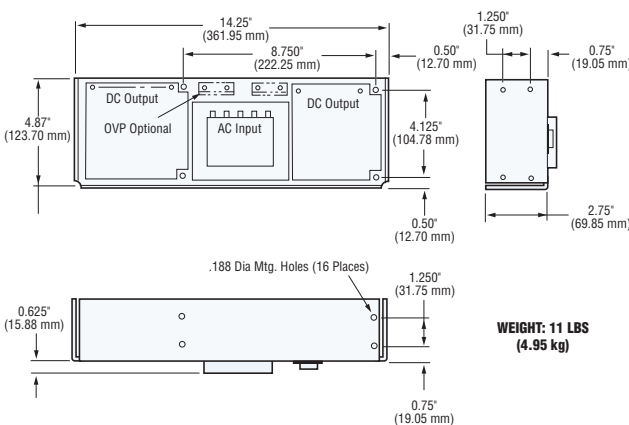
### E CASE

**OVERALL SIZE**  
14.00" x 4.87" x 3.53"  
(355.60 mm) x (123.70 mm) x (89.66 mm)



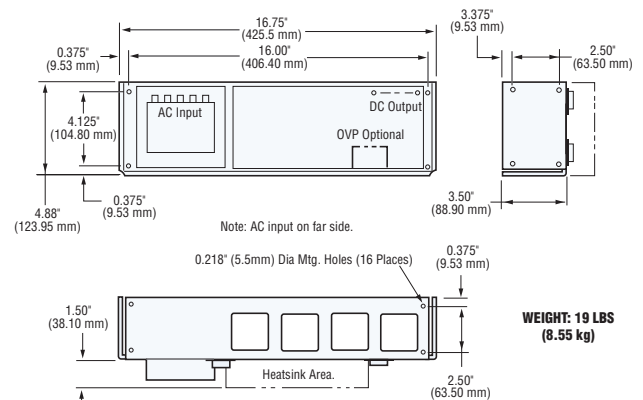
### DBB CASE

**OVERALL SIZE**  
14.25" x 4.87" x 3.38"  
(361.95 mm) x (123.70 mm) x (85.85 mm)



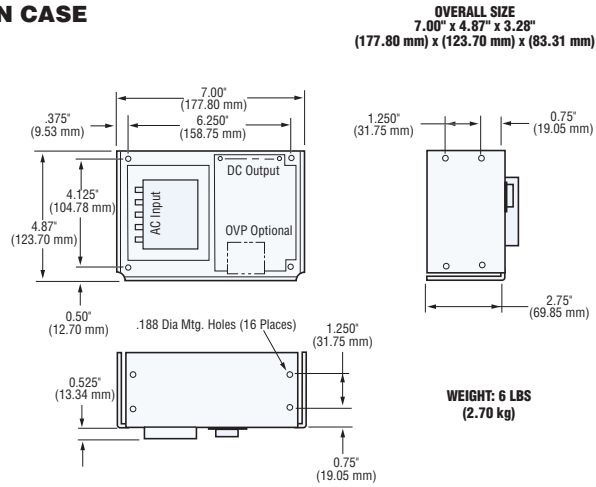
### F CASE

**OVERALL SIZE**  
16.75" x 4.88" x 5.00"  
(425.5 mm) x (123.95 mm) x (127.00 mm)

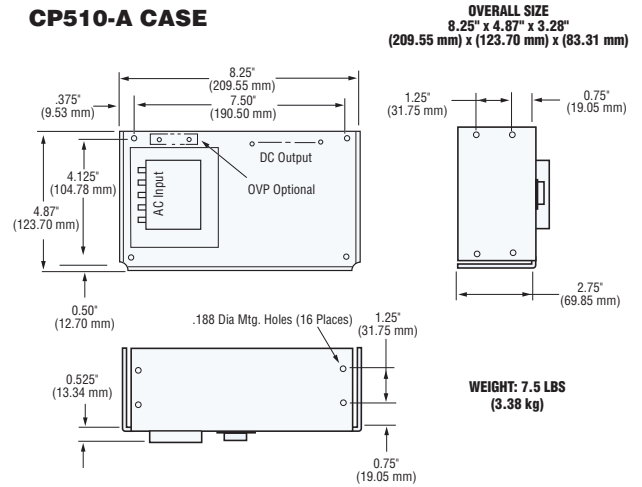


## INTERNATIONAL SERIES LINEARS

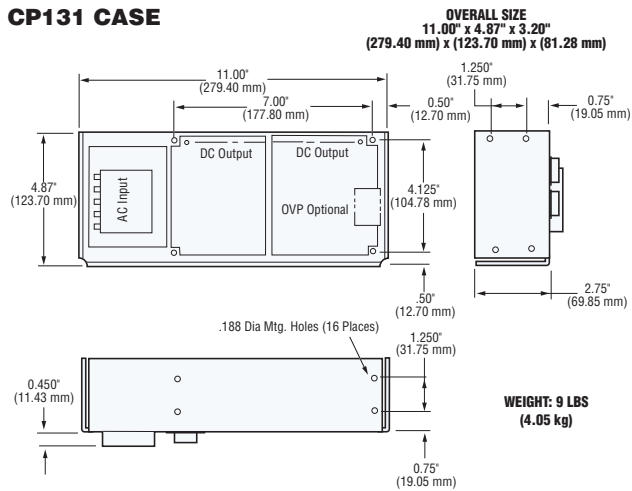
### N CASE



### CP510-A CASE



### CP131 CASE



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## **APPENDIX H**

### **Standard Operating Procedures**

# **STANDING OPERATING PROCEDURE**

**1310-0001**

**HYDROLYSATE PILOT PLANT  
BUILDING G-10**

**DECEMBER 2000**

**Royal Ordnance North America, Inc.  
Holston Army Ammunition Plant**

**STANDING OPERATING PROCEDURE  
FOR  
HYDROLYSATE PILOT PLANT, BUILDING G-10  
1310-0001**

**Issued: December 12, 2000**

**Prepared by:**

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12 Dec 00  
**Date**

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13 Dec 00  
**Date**

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**Manager Explosives**

13 Dec 00  
**Date**

Gail Plum  
**Safety Manager**

13 Dec 00  
**Date**

Signed  
**Environmental Manager**

13 Dec 00  
**Date**

Signed  
**Quality Assurance Manager**

13 Dec 00  
**Date**

Signed  
**Operations Manager**

13 Dec 00  
**Date**

**LOAD LIMITS**

<b><u>Building</u></b>	<b><u>Number of Personnel</u></b>	<b><u>Pounds of Class 1.1D Explosives</u></b>
G-10	Operational 4 Transient 4	500

The personnel and explosives load limits applicable to the current operations of Building G-10 must be conspicuously displayed on the outside of the building near the sign-in board.

**MSDS LISTING**

Material Safety Data Sheets for chemicals listed in this SOP are located in the Team Leaders Offices at Building 156, the Maintenance Team Leaders offices at Building 156, and in the Safety Office in Building 26.

<b><u>TITLE</u></b>	<b><u>MSDS NO.</u></b>
RDX	4304.0072
HMX	4801.0226
TNT	0599.1356
Tetryl	5795.
Composition B	4053.0072
Composition B-4	4070.0072
M1 Propellant	5794.
M8 Propellant	5793.
M28 Propellant	5776.
Sodium Hydroxide	2417.1824
Sulfuric Acid	0557.1831

Note: The purpose of operation of the Building G-10 Hydrolysis Pilot Plant is to support the Assembled Chemical Weapon Assessment (ACWA), which is focused on the evaluation of various technologies for eventual use in the destruction of chemical weapons and energetic materials contaminated with chemical weapons. The technology to be evaluated at Holston Army Ammunition Plant (HSAAP) involves the use of a caustic solution (sodium hydroxide) to hydrolyze energetic materials. All activities conducted in this program will be experimental in nature. The data and process parameters generated in the study being used to support the scale up and design of the Pueblo and Lexington Bluegrass Chemical Agent Disposal Facilities. In the work conducted at HSAAP, the hydrolysis technology will only be evaluated on energetic materials (i.e. select explosives and propellants). No chemical or biological weapons will be brought into the Holston AAP facility during this study.



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## I. INTRODUCTION

### A. Purpose

This procedure describes the building, equipment, and general operating processes used in the Hydrolysate Pilot Plant in Building G-10. Specific manufacturing instructions for the operation of the glass-lined reactor and its associated equipment is included in the “Normal Operation” section of this SOP.

### B. Process Description

Explosives or propellant are transported to Building G-10 from an assigned storage magazine. The material is initially staged on the third floor of Building G-10. The maximum amount of energetic material that can be transported to Building G-10 at any given time is 500 pounds.

The 2,000-gallon glass-lined reactor is filled with water to a level that is above the top agitator blade. The feed hopper on the third floor of Building G-10 is manually loaded with the appropriate energetic material (500 pounds maximum). Upon inspection of Building G-10 and implementation of the pre-operational checklist, all operators must leave Building G-10, and the reactor is remotely operated from the Building E-10 Control Room.

Sodium hydroxide is added to the reactor in a quantity required to achieve the targeted concentration of caustic. Using the weight-loss feeder, the energetic material is metered into the reactor at a specified feed rate. Upon addition of all energetic material, the reactor is held and closely monitored at relatively constant conditions for a specified time period to allow completion of the hydrolysis reaction (i.e., all explosives are destroyed). To monitor the effectiveness of the reaction, samples are remotely collected from the reactor throughout the reaction sequence.

Upon completion of the reaction, sulfuric acid is added in a sufficient quantity to neutralize the reactor contents (i.e., pH within a range of 5 – 9). The resulting Hydrolysate product is a concentrated salt solution consisting primarily of sodium sulfate. This Hydrolysate is pumped from the reactor to a designated storage tank in the Building G-10 Tank Farm.

## **II. SAFETY AND HEALTH CONSIDERATIONS**

### **A. Personal Safety Requirements**

#### **1. Personal Protective Clothing and Equipment**

1. Gloves, face shields, aprons, escape masks, safety glasses, safety shoes, flameproof clothing, respirators, and hearing protection are personal protective items available for the health and safety of production personnel and must be used when required.
2. Approved clothing for the Explosives Plant consists of, as a minimum, safety glasses or goggles with permanently affixed side shields, safety shoes, and a powder uniform which includes coveralls, cap, underwear and socks.
3. Flame retardant coveralls shall be worn in buildings or operations where there is exposure to open flames, the potential for flash fires or the possibility exists for contaminating clothing with materials that could flash.
4. Transient visitors may wear an approved protective smock and rubber or disposable, non-sparking overshoes during visits which do not exceed one hour. Rules concerning headwear, safety glasses/goggles and hearing protection must be observed.
5. Smocks or coveralls shall be completely buttoned at all times during visits into hazardous areas. Trouser cuffs must be turned under to preclude collection of explosives.
6. Hard hats must be worn where there is a reasonable probability of injury that can be prevented by such equipment.

#### **2. Precautions Necessary to Prevent Exposure to Hazardous Chemicals**

In processing explosives in the G-Buildings, the potential of exposure to chemicals and other hazardous materials is always present. Operators must take precautions to prevent such exposure.

- a. Process piping or pumps that may contain solvents or explosives should be flushed with water before they are disassembled. Valves used to isolate the particular section being worked on must be closed and tagged. Pumps must be de-energized and wiring disconnected. Use the "Lock and Tag Procedure" (PPS 6.20).
- b. When hand charging dry explosives to the hopper of the feeder, proper personal protective equipment should be worn by the operator, including gloves and respiratory protection as required for the particular explosive being processed.
- c. To prevent skin contact, gloves must be worn when taking samples.
- d. Before entering process vessels, ensure that the vessels are empty of all explosives residue and have been boiled out with water to

remove any solvent vapors. The vessel atmosphere must be tested by the Safety Department representative, and a Safety Permit for confined space entry/work must be complied with.

3. Control Measures to be Taken if Contact With a Hazardous Chemical Occurs

To minimize burns or irritation caused by contact with hazardous chemicals, the affected individual should:

- a. Flush the affected area with water. Emergency showers should be used when the chemical contamination affects a large portion of the body.
- b. Flush with water for 15 minutes. Immediate use of an emergency shower followed by the use of a shower at the change house is acceptable.
- c. Remove clothing from the affected area as soon as possible. Wrap body in a safety blanket after showering at the site or dress in clean clothing.
- d. Obtain medical attention by first contacting Emergency Treatment Personnel (EMTs), providing information about the particular chemical involved. Seek additional medical treatment as needed or as recommended by EMT personnel.

4. Procedures for Safely Opening Process Equipment

a. General

Normally, all process vessels and lines associated with the manufacturing operations in Building G-10 should remain closed. Atmospheric vents provide pressure relief on chemical tanks or the explosive manufacturing vessels. Necessary precautions must be taken to protect operator(s) from chemical exposure when opening any process vessel.

b. Explosive Processing Vessels

Samples are taken from access ports on the processing vessels. These sampling ports may only be opened if the ventilation system is operating. Caution should be exercised to crack the port to check for fumes before opening it all the way. Operators should minimize this operation and take precautions to avoid inhaling fumes for prolonged periods and should wear rubber gloves to avoid skin contact.

c. Process Piping and Pumps

Process piping and pumps should never be opened by operators. Manufacturing Maintenance personnel only are authorized to disconnect flanges in explosives service. Operators do have the responsibility to ensure that mechanics are not exposed to explosive or chemicals when breaking lines or opening pumps.

Piping, pumps or other equipment should be flushed with water before allowing maintenance forces to perform maintenance. In addition, operators are responsible for initiating appropriate Safety Permit (PPS 1.20) and following the "Lock and Tag Procedure" (PPS 6.20) prior to initiating this type of maintenance activity.

## B. Material Safety and Quality Requirements

### 1. Quality Control Measures

Operation of the hydrolysate pilot plant in Building G-10 will initially be conducted under the guidance of chemists and engineers to establish the optimum conditions for the pilot equipment. The resultant hydrolysate will be sampled to determine the effectiveness of the pilot process in destroying the RDX or HMX present in the feed to the reactor. Material safety and quality requirements will be established based upon these initial operations and will be incorporated into this SOP as appropriate.

### 2. Environmental Treatment, Storage, Disposal, and Accidental Discharge Instructions

Accidental discharges of chemicals during operation of the pilot plant will be reported immediately by contacting the WSI Security Dispatcher by dialing 9-1-1. When reporting an accidental discharge, provide as much information as possible to the emergency response crew to expedite control of the discharge. Include the chemicals discharged and the estimated quantity. If possible, take appropriate actions to stop further discharge and terminate operations until the discharge is brought under control and cleanup efforts are completed. Clean-up of the discharged material will begin as soon as the site is deemed safe by the person in charge of the Command Post. Operators and line supervision will cooperate fully with emergency response personnel to clean up discharged material and check for potentially hazardous contamination.

#### a. Equipment Designation and Safety Functions

##### 1) Utilities

##### a. Electrical and Back-up System

High line voltage distribution is 13.5 KV, which feeds into a step-down transformer. Three-phase, 480 volts feeds into the building control room then goes into the switchgear. The switchgear feeds three-phase, 480 volts to the pump motors and agitators. The switchgear also feeds the lighting transformer for all of the building lighting.

A backup transformer is fed from another source, which is for emergency power only. There is a transfer switch between normal power and emergency power. This switch will operate when normal power is lost and will automatically switch to emergency power.

The back-up power will support operation of the following equipment: dissolver agitators, still agitators, slurry pumps, solvent pumps, vacuum pumps, and the building lighting system. No other equipment should be operated while back-up power is in use. If too many motors are started, the system might become overloaded, causing the back-up power system to fail, leaving the building without any power.

In the event of a primary power failure, a transfer station will automatically bring the back-up power supply on-line. The switchover normally takes 1-5 seconds. When primary power is restored, the transfer station will automatically switch back to the primary supply. The transition from back-up to primary power is almost instantaneous.

In order to determine which power supply (primary or back-up) is being used; observe the indicator lights at the transfer station. There are two indicator lights mounted on the center control panel. When the green light is on, the primary power is in use. The tag above this light reads "LOAD CONNECTED TO NORMAL." When the red light is on, the back-up power supply is in use and the tag above this light will read "LOAD CONNECTED TO EMERGENCY".

Maintenance on any electrical equipment must be performed by Electrical Department personnel who keep the only keys to the electrical control room door lock. It is the responsibility of the building operators to ensure the breaker(s) of an electrical circuit is properly disconnected, locked and tagged. Operators are also responsible for initiating appropriate Safety Permits (PPS 1.20) and following the "Lock and Tag Procedure" (PPS 6.20) prior to initiating this type of maintenance activity.

b. Steam System

Steam is produced at the Building 200 Steam Plant, and is transported as 300 psig superheated steam to operating buildings. Building process steam is reduced, controlled and passed through the automatic valve at normally 15 psig (Maximum 38 psig). When the steam passes through the automatic valve, it is directed through a steam header, which has a pressure relief valve set at 45 psig. The header also has a desuperheater system where water is injected to desuperheat the steam to ensure that explosives are maintained at temperatures safely below their detonation temperature. If the steam temperature exceeds 142 degrees Centigrade, the steam valve will be cut off by a high temperature control switch.

Steam is taken from the 300 psig superheated steam supply and reduced to 15 psig. This product is supplied to bayonet heaters in the storage tanks, to the boxway steam, tracer lines, steam for the clean-up stations, and for the building heating steam.

c. Water Systems

1) Cooling Water:

River water enters the building through a 12-inch line and is filtered as it flows through a twin basket strainer. Only one side of the strainer is used at a time. When a one basket becomes dirty and/or the water pressure drops below 25 psig, the system will automatically backwash (flush) and clean the dirty strainer. This process is repetitious.

2) Filtered Water:

A stainless steel (multiple tube), can-type filter is installed on the filtered water supply line to re-filter the incoming water from the plant-wide filtered water system.

3) City Water:

City water is used for drinking and sanitation purposes with Building G-10.

2) Safety Equipment

3) Process Systems and Equipment

### III. SEQUENCE OF OPERATIONS

A. Initial Start-up

1. Preliminary System/Equipment/Control Checks

1. Tank Farm:

- 1) Manually close the valves (identified as HV-10, HV-12, HV-14, and HV-15) on the discharge lines of Tank Nos. GT-10, GT-12, GT-14, and GT-15 that are designated for storage of the hydrolysate product.
- 2) Manually open the valve (identified as No. HV-21-1) located on the discharge line of the GT-11 Caustic Storage Tank.
- 3) Manually close (or confirm closed) the valve (identified as HV-22-1) located on the discharge line of the GT-13 Acid Storage Tank.



- 4) Manually close the two dike drain valves located on the north side of the two diked areas in the Building G-10 Tank Farm using a tee-handle.
- 5) Check the temperature recorders on Tank Nos. GT-10, GT-11, GT-12, GT-14, and GT-15 and record the tank temperatures in the log.
- 6) Using a graduated dipstick (in inches), measure the tank levels in all hydrolysate storage tanks (Tank Nos. GT-10, GT-12, GT-14, and GT-15). Log all tank levels into the logbook.
- 7) If upon visual inspection, liquid is visible inside the diked area check then inform supervision who will determine whether the liquid is the result of a leaking tank or simply water from rainfall or steam condensate. If any observed liquid is determined to be simply rainfall or steam condensate then open the two dike drain valves to drain water to the industrial sewer. Close the valves immediately upon completion of this operation.

2. Inside Building G-10:

- 1) Manually open (or confirm open) the valve (identified as No. FV-1 and located on the third floor {west side} of Building G-10 adjacent to the material lift bay) which supplies water to the system deluge nozzle above the feed hopper.
- 2) Isolate the sampler to prevent remote operation by switching the SAMPLER E-STOP lever to the off position. The E-STOP lever is located on the west wall adjacent to the sampler in Building G-10. The remote sampling cabinet can now be prepared as follows:
  - Close the drain valve located on the ice water bin in the sample cabinet
  - Remove the screen from the sample tray in the sample cabinet.
  - Load the sample bin to the top of the screen support with ice, and replace the screen.
  - Install the four latches to secure the screen.
  - Place ice in the center compartment on top of the screen.
  - Using the water hose mounted on Column C-3 in the west side of Building G-10, manually add water to the sample bin filled with ice until the level reaches indicator mark approximately one inch from the top lip of the ice bin.

- Load the new sample bottles, containing the pre-measured acid heel for quenching, into the sample carousel. (Make sure all bottles are threaded snugly into the Teflon bottle holders.)
  - Using the manual crank, lift the water bath to its maximum height, which is at an appropriate level to partially cover the sample bottles in the ice water.
  - Close the door(s) of the sample cabinet and secure with the latches.
  - Switch the SAMPLER E-STOP lever to the ON position to activate the sampler thereby enabling computer control.
- 3) Manually open the two valves (identified as Nos. 201-A and 201-B) on the line providing cooling water to the condenser located on the north side of the reactor skid (upper level) opposite of the manhole cover.
  - 4) Manually open the valve (identified as No. A-1) installed in series with the automated valve on the acid feed line located on the east side of the reactor skid (upper level).
  - 5) Manually open the valve (identified as No. C-1) installed in series with the automated valve on the caustic feed line located on the east side of the reactor skid (upper level).
  - 6) Manually open the air sample valve (identified as No. 100) on the port on the gas sampling valve located on the east side of the reactor skid (upper level).
  - 7) Manually close the valve (identified as No. 23-1) on the pipe allowing dump tank contents to be pumped back to the reactor (located on the east side of the reactor skid (upper level) adjacent to the manhole cover).
  - 8) Manually open the valve (identified as AS-1) providing water to the water seal of the agitator located on the south side of the reactor skid (upper level) next to the manhole cover. The flow of water should be adjusted to achieve maximum flow without overflowing the seal. Open the manhole cover and visually confirm that water is not leaking into the reactor. Also, check the discharge pipe (labeled as Agitator Seal Water) at the bottom of the reactor skid (north side) to ensure water is flowing.
  - 9) Manually open the valve (identified as No. R-1) on the circulation loop of the reactor that is located adjacent to the manhole on the reactor.

- 10) Manually open the valve (identified as No. 201-C) on the line providing cooling water to the heat exchanger located on the north side of the reactor skid (lower level) opposite of the manhole cover.
- 11) Manually open (or confirm opened) the valve (identified as No. ST-2) supplying steam to the tracer line on Sample Valve No. 100 located on the north side of the reactor skid (lower level).
- 12) Manually open the valve (identified as ST-1) on the line supplying steam to the reactor heat exchanger located on the north side of the reactor skid (lower level).
- 13) Manually open the valve (identified as No. 201-D) on the line allowing flow of water from the jacket on the reactor to the industrial sewer located on the west side of the reactor skid (lower level).
- 14) Manually close (or confirm closed) the valve (identified as No. HV-7-1) on the recirculation pump suction line, located on the south side of the reactor skid at ground level. (This valve will later be used to collect the final sample of hydrolysate product at the end of a reactor run.)
- 15) On the discharge side of the hydrolysate transfer pump (No. PU4), manually close Valve Nos. 19-1, HV-42, and HV-12-2. Also, at this position, manually open Valve No. HV-12-1 on the overflow line between the reactor and the dump tank.
- 16) Manually open the valve (identified as No. ST-3), located adjacent to Valve No. 19-1 at floor level, that supplies steam to the steam tracer line on the gas sampling line coming from the reactor.
- 17) Inspect the water level in the dump tank. If necessary, adjust the level to the top of the interior heating / cooling coils (approximately 2000 gallons).
- 18) If water needs to be added, open the valve (identified as No. W-1) on filter water inlet line to the dump tank (located on the west side of the dump tank adjacent to the manhole).
- 19) Manually open the valve (identified as No. AS-2) providing water to the water seal on the dump tank (located on the west side of the top of the dump tank). Visually confirm water flow by inspecting the drain line outlet in the drain at the north wall at the back of the dump tank.

- 20) Manually open the scrubber water valve (identified as No. SW-1) and set the water flow at 40 gallon per minute (gpm)  $\pm$  5 gpm.
- 21) Manually open the valve (identified as No. HV-41) located on the overflow line (first floor) between the reactor and the dump tank.
- 22) Turn on the river water flow valve (identified as 10-3) using handspring switch No. HS 10-3 (located on the first floor on the north side of the dump tank). The hand – spring switch No. HS 10-3 should be in the “Off” (“down”) position.
- 23) Turn off hand-switch valve No. HS 10-2 (i.e. switch in down position) located on the first floor on the south side of the dump tank. Valve No. 10-2 switches between steam-on the water and out of the dump tank heating / cooling coils.
- 24) Manually close (or confirm closed) the valve (identified as No. D-2), which allows liquid within the diked area around the reactor to be drained to the dump tank, located on the south side of the dump tank (first floor).
- 25) Manually close (or confirmed closed) the valve (identified as No. D-1) located on the bottom of the dump tank that controls the flow of dump tank contents.
- 26) Manually close Valve No. C-2 and manually open Valve Nos. C-3 and C-4, which control the direction of flow in the caustic transfer lines, located on the first floor (west side) of Building G-10.

### 3. Building E-10 Control Room:

Note: the pre-operational activities in E-10 can be undertaken whilst Building G-10 is manned.

- 1) Switch on the computer and log in.
- 2) Adjust the remote feeder set points to feed the reactor with the desired quantity of energetic material (maximum 500 LB) at the desired feed rate as specified on the batch sheet.
- 3) Enter the desired reaction digest time into the PLC via the computer.
- 4) Click on the “START BATCH” icon in the computer screen to initiate a batch startup.
- 5) Check to ensure that the reactor tank level is less than 10% (which could mean zero) and that the measured temperature

inside the reactor is less than 90°C. If either of these conditions is not true then consult with supervision.

- 6) Add water to the reactor (quantity as specified on the batch sheet). OPTIONAL: Apply heating to the reactor, set point  $80\pm 2^{\circ}\text{C}$  unless otherwise specified on the batch sheet.

2. Fire Prevention Systems:

Building G-10 is equipped with a dry-pipe sprinkler system (except in the area above the weigh feeder), which can be activated from paddles located at each exit door from the building. The weigh feeder is equipped with a wet pipe system. Dry chemical fire extinguishers are available on all floors to be used to extinguish fires that do not involve explosives.

**IMPORTANT: UNDER NO CIRCUMSTANCES WILL AN OPERATOR ATTEMPT TO EXTINGUISH A FIRE, WHICH MAY INVOLVE EXPLOSIVES. PERSONNEL SHALL LEAVE THE BUILDING IMMEDIATELY USING AS MUCH PROTECTIVE COVER AS POSSIBLE. PERSONNEL SHALL ACTIVATE DELUGE SYSTEMS AND FIRE ALARM EQUIPMENT WHILE ESCAPING.**

3. Emergency Alarms:

Each exit from the building has an emergency alarm lever located on the right side. In the event of an emergency, the first person noticing the emergency should press the emergency alarm lever(s), thus notifying other building occupants of the emergency. The alarm will be a continuous howling of a horn. Activation of this alarm will automatically shut off the steam to the building. The alarm may be stopped by depressing the stop button located outside the electrical control room door. Emergency alarms must be tested a minimum of every two (2) months for reliability and adequacy.

4. Starting Utilities Systems:

5. Receiving Process Chemicals:

a. Sodium Hydroxide:

Sodium hydroxide within an approximate concentration range of 20 – 50% is stored in Tank No. GT-11 in the Building G-10 Tank Farm. The maximum storage capacity of the tank is about 6,000 gallons. In operation of the pilot facility, the sodium hydroxide is pumped directly into the hydrolysis reactor from the storage tank. The sodium hydroxide is diluted in the reactor (if necessary) to the desired caustic strength for the reaction.

b. Sulfuric Acid:

Concentrated sulfuric acid (approximately 94%) is stored in Tank No. GT-13 in the Building G-10 Tank Farm. The maximum storage capacity of the tank is about 6,000 gallons. The sulfuric acid is used to neutralize the contents of the reactor upon completion of the hydrolysis reaction. In this operation, the acid is pumped directly into the reactor from the storage tank.

c. Energetic Materials:

On an “as needed” basis for the program’s experimental runs, energetic material will be transported to Building G-10 (in maximum quantities of 500 lbs. at a time) using a tractor-trailer. Materials handling functions associated with the transport of energetic materials to Building G-10 will follow standard procedures outlined in Standing Operating Procedure No. 1700-9600.

- 1) Visually inspect all labels on material storage boxes at Magazine No. 100, prior to loading trailer, to ensure that the proper material is being transported to Building G-10.
- 2) Secure the trailer containing the energetic materials at the loading dock on the south side of Building G-10 using standard procedures outlined in RONA SOP No. ???
- 3) Using a pallet jack, transport the pallet containing the energetic material onto the Building G-10 material lift, and shut all doors on the material lift.
- 4) Walk to the third floor via stairs.
- 5) Call the material lift to the third floor using the control button.
- 6) Using the water hose mounted on the wall adjacent to the weight-loss feeder, thoroughly wet the floor area around the feeder area.
- 7) Using the pallet jack, transport the pallet to the marked staging area on the floor adjacent to the weight-loss feeder.

B. Normal Operations:

1. Charging Energetic Materials into the Hopper on the Weight-Loss Feeder:

Note: All activities associated with the charging of energetic materials into the feed hopper will require Personal Protective Equipment (PPE) to be worn including safety glasses, gloves, and a respirator / dust mask.

- a. Activate the Red E-Stop Button (by pressing in) located adjacent to the stairway on the work platform to completely disable the weight-loss feeder.

- b. Remove the solid lid from the feed hopper.
  - c. Place the lid out of the immediate work area on the left side of the platform. Use the available ground strap with clip to ground the lid.
  - d. Visually inspect the interior of the hopper for contamination.
  - e. Ensure the metal grid ( $\frac{3}{4}$  inch openings) is in place over the mouth of the hopper.
  - f. Manually carry one 50 lbs. box of energetic material to the worktable on the platform.
  - g. Open the lid of the box and collect a representative sample of the material (about 50 grams) into a fiber sample carton (quart-size). Retain as file sample, to be disposed of at the end of the HYDROLYSIS Program or by December 2002, whichever occurs first.
  - h. Manually dump the contents of the box (through the grid screen) into the hopper.
  - i. If necessary, manually stir any material collected on the grid screen to facilitate flow into the hopper.
  - j. Manually collect any pellets of energetic material on the grid screen, which will not flow, into the hopper. This material should be placed into an approved explosive waste container and disposed as outlined in the G-10 SOP No.
  - k. Dispose of any explosive / propellant contaminated trash in the trash bin provided at the work platform.
  - l. Repeat Steps f -k until all boxes on the skid have been loaded into the hopper (maximum of 10 boxes or 500 lb of material, whichever is the greater).
  - m. After charging all of the available energetic material into the hopper, clean and dispose of all trash and debris around the work platform.
  - n. Using the water hose, thoroughly wash the work platform and the area around the weight-loss feeder. Do not wash down the weight-loss feeder itself.
  - o. Visually inspect the outlet of the bottom screw on the weight-loss feeder and the outlet pipe feeding the reactor to ensure no blockage.
  - p. Visually inspect the cloth chute between the outlet of the weight-loss feeder and the energetic feed pipe into the reactor to ensure proper placement.
2. Preparation of Building G-10 for Unmanned Operations:
- a. Inspect the reactor to ensure that the filtered water addition is proceeding or has been completed and that the reactor and associated equipment appears in good order (no unusual liquid discharges from pumps or process lines).
  - b. Ensure that all exits on the first floor are secured, with the exception of the main exit.
  - c. The final activity that must be completed in Building G-10 is to return to the 3<sup>rd</sup> floor of Building G-10 and re-activate the Red

Feeder E-Stop button (by pulling out). Once complete, no other operations or inspections are permitted in Building G-10.

- d. When leaving the building, firmly secure the door so that the door interlock is activated. Return to Building E-10.

3. Operation of the Hydrolysis Reactor:

Note: the following operations must be carried out remotely.

- a. Start agitator, speed  $100 \pm 1$  RPM unless otherwise specified on the batch sheet.
- b. Start recirculation pump. Note: the pump should self-prime in approximately ten minutes. If flow within the recirculation line is less than 200 LB / minute after ten minutes, then refer to supervision.
- c. If specified on the batch sheet, take a sample of filtered water from the reactor using the remote sampler.
- d. Add the specified quantity of sodium hydroxide (caustic) to the reactor. Allow to mix for a minimum of two minutes after the final addition of caustic.
- e. If specified on the batch sheet, take a sample of the caustic solution from the reactor using the remote sampler.
- f. Allow the reactor to heat up to the temperature specified on the batch sheet (if necessary, adjust the reactor heating accordingly).
- g. Start the energetic feeder.
- h. After the final addition of energetic material, remotely flush the feeder with five gallons of filtered water to remove residual energetic material from the feeder and associated reactor piping.
- i. Fifteen minutes after the final addition of energetic material has been made to the reactor, Building G-10 can be manned if required, with approval from supervision.
- j. Monitor process via computer / PLC and report any unusual occurrences to supervision.
- k. If the actual temperature of the reactor exceeds  $3^{\circ}\text{C}$  above the set point temperature then notify supervision.
- l. Take samples at the appropriate time as specified on the batch sheet.
- m. After the reaction is complete, remove the sample bottles from the remote sampling cabinet in Building G-10. Submit the samples to the appropriate personnel (as directed by supervision) for processing and packaging.



- n. At the end of the reaction, after the reactor contents have cooled to less than  $35 \pm 5^\circ\text{C}$ , start the neutralization step (i.e. the addition of sulfuric acid). The targeted pH of the neutralized hydrolysate is within a range of 5.0 – 9.0.
- o. After neutralization of the reactor contents to the appropriate pH, fill two, clean two-gallon sample bottles with hydrolysate by carefully opening valve identified as No. HV-7-1 on the recirculation pump suction line, located on the south side of the reactor skid at ground level. The second two-gallon sample is to be kept as the process sample. The first sample is to be added either to the reactor or to a hydrolysate storage tank, as specified by supervision.
- p. Print off copies of the process data as specified on the batch sheet.
- q. Open / close valve xyz ready for transferring hydrolysate to tank farm (tank number as specified by supervision).
- r. Transfer hydrolysate to the storage tank using the reactor computer controller in Building E-10.

4. Clean-up of the Reactor:

- a. Add sufficient filtered water to the reactor to enable the recirculation pump to start.
- b. Start the recirculation pump, agitator (speed  $150 \pm 10$  RPM) and water spray nozzle. Mix / recirculate for five minutes. (Note: recirculation pump will require approximately ten minutes to self-prime).
- c. Open the reactor manhole cover and wash down the interior of the reactor using the water hose adjacent to the reactor.
- d. Pump reactor contents to the hydrolysate storage tank.
- e. Repeat steps 7.1 to 7.4 for a second time.
- f. Flush / blow out hydrolysate lines.

C. Operation Shutdown:

1. Tank Farm:

- a. Manually close or confirm close all valves on caustic, acid, and hydrolysate storage tanks (Tank Nos. GT-10, GT-11, GT-12, GT-13, GT-14, and GT-15).
- b. Close (or confirm closed) the two dike drain valves located on the north side of the two diked areas in the Building G-10 Tank Farm using a tee-handle.
- c. Check the temperature recorders on Tank Nos. GT-10, GT-11, GT-12, GT-14, and GT-15 and record the tank temperatures in the log.

- d. For all hydrolysate storage tanks to which material had been pumped during the day's operation, use a graduated dipstick to measure the level in the tank(s) and record the data in the logbook.

2. Inside Building G-10:

- a. The shutdown procedure on the remote sample cabinet should be as follows:
  - 1) Isolate the sampler to prevent remote operation by switching the SAMPLER E-STOP lever to the off position.
  - 2) Place the hose on the drain valve on the ice water bin in the sampler cabinet.
  - 3) Open this valve and let the water in the cabinet to flow into the diked area.
  - 4) Leave the drain valve open, and the two doors unlatched and open during the night shift in which the sampler will be inactive.
- b. Manually close Valve No. 201-B, to close the flow of river / cooling water to the condenser, located on the north side of the reactor skid (upper level) opposite of the manhole cover.
- c. Manually close Valve No. A-1 installed in series with the automated valve on the acid feed line located on the east side of the reactor skid (upper level).
- d. Manually close Valve No. C-1 installed in series with the automated valve on the caustic feed line located on the east side of the reactor skid (upper level).
- e. Valve No. AS-1, which provides water to the water seal on the reactor agitator, should be left open to maintain the flow of water on the seal. However, if operations are not planned for the reactor for several days, then the Valve No AS-1 should be closed to conserve water.
- f. Manually close Valve No. 201-C on the line that provides cooling water to the heat exchanger and is located on the north side of the reactor skid (lower level) opposite of the manhole cover.
- g. Manually close the Steam Valve No. ST-2 on the line that supplies steam to the reactor heat exchanger and is located on the north side of the reactor skid (lower level).
- h. Confirm that Valve No. HV-12-1, located on the overflow line between the reactor and the dump tank, is open.
- i. Manually close Valve No. AS-2 providing water to the water seal on the dump tank.
- j. Manually close Valve No. SW-1 supplying water to the scrubber system.

- k. Turn off the flow of river water to the cooling coils on the dump tank by activating handspring valve No. HS 10-3 located on the north side (first floor) of the dump tank. The handspring valve should be in the "On" (up) position.
- l. Confirm that Valve No. D-1, located on the bottom of the dump tank, is closed.
- m. Manually close (or confirm closed) Valve Nos. C-2, C-3, and C-4, which control the direction of flow in the caustic, transfer lines and are located on the first floor (west side) of Building G-10.

D. Emergency Shutdown Operations:

1. Fire or Explosion:

Training must stress the safety of the individuals and their co-workers in both fire and explosion situations. Employees should understand that their first obligation is to alert fellow workers, assure his/her own safety and then to immediately report the emergency to the Fire Department (9-1-1). Only after this has been done and only when a fire involves non-explosive material, should an effort be made to extinguish the blaze. When explosions occur, no action should be taken to control processes.

If a fire occurs and does not involve explosives and if you believe you can bring it under control, immediately warn all building occupants. Once this is done, report the fire by telephone, radio or messenger. The operation must be shutdown. If the fire is small and is away from explosives, an effort may be made to extinguish the fire using available fire extinguishers. If the fire cannot be quickly contained with reasonable safety to yourself, activate the deluge system and emergency alarm as you exit the building.

Operations personnel may be requested by the Command Post to assist in providing information necessary to deal with the emergency. Report to your Team Leader and be available to assist.

**NOTE: UNDER NO CIRCUMSTANCES WILL ANYONE ATTEMPT TO FIGHT FIRES INVOLVING EXPLOSIVES EXCEPT TO MANUALLY ACTIVATE INSTALLED FIRE EXTINGUISHING EQUIPMENT. PERSONNEL SHALL LEAVE THE BUILDING IMMEDIATELY USING AS MUCH PROTECTIVE COVER AS POSSIBLE. PERSONNEL SHALL ACTIVATE DELUGE SYSTEMS AND FIRE ALARM EQUIPMENT WHILE ESCAPING.**

If an explosion occurs in an explosive operation, the operator(s) in the affected building will take the first and initial steps that are vital to personnel and/or damage to property. Activate the emergency alarm and deluge system while exiting the building. Contact emergency response teams by calling 9-1-1. **NOTE: DO NOT HANG UP**

**UNTIL YOU ARE CERTAIN INFORMATION CONCERNING THE INCIDENT HAS BEEN RECEIVED BY EMERGENCY RESPONSE PERSONNEL.**

Notify your Team Leader of your whereabouts and if you are injured. Make yourself available to assist as requested by the Command Post.

If the explosion results in a fire, do not attempt to fight the fire. Evacuate the building immediately and follow instructions as provided above to ensure your safety, and to communicate details of the incident to emergency response personnel.

2. Accidental Discharge of Chemicals or Explosives:

In the event of a chemical or explosive discharge, instructions in the Installation Spill Contingency Plan will be followed. The operator will report all accidental discharges immediately to the Team Leader and the Safety Department (9-1-1) giving the name of the chemical discharged, time of the discharge, approximate amount, and location of the discharge. Similarly, Environmental Affairs will be notified to allow notification of applicable Local and State agencies, and to provide assistance in containment/cleanup of the discharged material. Government (HSAAP) officials will also be notified immediately.

3. Aborted Operations:

The hydrolysis reactor can be shut down by the operator at any time during the experimental run by activating the “Dump” icon on the PLC control panel. This “Dump” of the contents of the reactor and abort of the experiment will automatically trigger a number of inherent safety features of the reactor system to include:

- a. The contents of the reactor will be pumped via both the discharge pump and recirculation pump into the dump tank using independent lines.
- b. The filtered water inlet valve will be opened to flood the reactor with 2,000 gallons of cold filtered water.
- c. The explosive feed will automatically shut down (if in operation).
- d. Maximum cooling water will automatically be fed from the heat exchanger to the reactor.
- e. The agitator on the reactor will switch on (if not all ready running).
- f. The agitator on the dump tank will switch on (if not all ready running).

E. Temporary Operations:

1. In certain cases process idling is permitted to replace equipment of a critical nature. In case of utility problems or failure, accidental discharge, severe weather conditions or in any circumstance where

idling the building is the safe and proper action to take. Process idling is accomplished by turning off the steam to all process systems. All agitation is left intact and any transfers to and from the G-Building are stopped.

2. Building equipment will be cleaned and maintained on a regular basis. At the start of each shift, operators will visually inspect the building equipment to check if the equipment is clean. Steam and water will be used to clean any contaminated equipment.

F. Start-up Following Emergency Shutdown:

Following an emergency process shut-down, and correction of the process / facility fault that caused the emergency, the following actions need to be undertaken:

1. Transfer the contents of the DUMP TANK (referred to hereafter as reaction mixture) to one of the following locations (decision to be made by Operations management – Explosives Division Manager or Process Engineer)
  - a. Hydrolysis Storage Tank
  - b. Another recrystallization still in Building G-10
2. Pre-heat the reactor to 85°C.
3. Pump 1250 gallons of the reaction mixture to the reactor.
4. Switch on recirculation pump and agitator. Mix until contents up to temperature ( $85 \pm 2^{\circ}\text{C}$ ).
5. Add filtered water or caustic solution to bring caustic concentration to 12%.
6. Stir reactor contents for 6 hours to complete the hydrolysis reaction.
7. Cool the reactor contents to contents to  $35 \pm 2^{\circ}\text{C}$ .
8. Neutralize and process as for a normal hydrolysate experiment.
9. Transfer hydrolysate to the hydrolysate storage tanks in the G-10 tank farm.
10. Repeat steps 3 through 9 until all 'DUMP TANK' reaction mixture has been processed.
11. Shut down process and clean up as per SOP.

## **APPENDIX I**

### **Failure Mode and Effects Analysis, Initial**



ROYAL ORDNANCE NORTH AMERICA, Inc.

**HOLSTON ARMY AMMUNITION PLANT (HSAAP)**  
**BUILDING G-10 ENERGETIC MATERIAL**  
**HYDROLYSIS FACILITY FAILURE MODE**  
**EVALUATION REPORT**

Compiled by:

\_\_\_\_\_  
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## **CHANGE RECORD**

Issue Status

Affected pages

A

Document issued to enable live G-10 facility commissioning

Note: Amendments to this document will be achieved by re-issuing the entire document.



## **1. INTRODUCTION**

- 1.1. This document describes the failure modes evaluated in the new Holston Army Ammunition Plant Building G-10 Energetic Material Hydrolysis facility ('HYDROLYSIS FACILITY').
- 1.2. The failure modes were tested as part of the inert facility commissioning experiments during November / December 2000, a pre-requisite of live commissioning trials.
- 1.3. The failure modes were principally evaluated to verify the correct operation of the HYDROLYSIS FACILITY in the event of a given single failure mode.

## **2. HYDROLYSIS FACILITY**

- 2.1. A detailed description of the HYDROLYSIS FACILITY can be obtained by referring to the facility layout drawing (XYZ). A summary of the features of the HYDROLYSIS FACILITY are given below:
- 2.2. The nucleus of the HYDROLYSIS FACILITY consists of a 2,000-gallon glass-lined reactor (Pfaudler design), located in the center bay on the second floor of Building G-10. The reactor is fitted with comprehensive instrumentation:
  - 2.2.1. Volumetric flow meter devices fitted to all liquid feed ports (3).
  - 2.2.2. PH sensor (1).
  - 2.2.3. Redundant temperature probes in dip-tubes located in the reactor (2)
  - 2.2.4. Recirculation line with independent pump.
  - 2.2.5. Mass flow meter, fitted into the recirculation loop, with an additional option for indicating temperature or liquid density (1).
  - 2.2.6. Variable speed agitator.
  - 2.2.7. Pressurized heating / cooling thermocirculator (Temperature range nominally 30-100°C).
  - 2.2.8. Independent discharge pump for emptying the reactor.
- 2.3. The reactor can be fed solid feeds (energetic or inert) using an Acrison loss in weight feeder, located on the third floor of Building G-10 directly above the reactor. The feeder is designed to feed material at controlled feed rates up to 500 LB / hour. The feed hopper capacity is 500 LB (nominal).
- 2.4. The reactor is piped directly to an existing, previously unused Holston recrystallization still (nominal 6,000 gallon capacity). The still is located on the second floor, but a significant portion of the still volume is located under the second floor; the still is thus

“below” the glass lined reactor in terms of height. This enables the use of gravity flow between the reactor and the still, if required.

- 2.5. The still is designed to act as a highly capable secondary storage and processing tank for the contents of the reactor in the event of their being a reaction runaway or other major operational issue associated with the reactor. The still is termed ‘DUMP TANK’ for this reason, which term ‘DUMP TANK’ is used hereafter in this document. The contents of the reactor can be pumped or filled by gravity to the ‘DUMP TANK’ by three independent means (described in detail in a later section).
- 2.6. A remote sampling device is also fitted to the reactor to enable samples to be remotely taken from the reactor recirculation line during a reaction. This enables a reaction to be monitored. A maximum of sixteen (16) samples can be remotely obtained per experiment. The sample bottles can be pre-filled with a reaction-quenching agent and/or flushed with quenching after being filled with a sample (remotely programmable). The samples can also be suspended in ice-cold water throughout an experiment (thereby aiding reaction quenching).

### **3. FAILURE MODE EVALUATION**

- 3.1. The failure modes evaluated relate to the critical operating parameters and failure modes identified in the conceptual Failure Mode and Effects Analysis conducted on the HYDROLYSIS FACILITY during CY 2000<sup>1</sup>.
- 3.2. The failure modes investigated and the results obtained are as shown in Table 1.
- 3.3. For each failure mode, an assessment has been made of the response of the equipment (PLC, software, reactor, feeder, dump-tank etc.). This assessment is summarized in Table 1 for each failure mode.
- 3.4. In addition to the failure mode experimental trials conducted on the HYDROLYSIS FACILITY, a Failure Modes and Effects Analysis (FMEA) has been conducted on the hydrolysis process pre-operational checks as defined in the Hydrolysis Process Manufacturing Instructions. The FMEA was conducted in accordance with the FMEA technique defined in the earlier FMEA report<sup>1</sup>. The Manufacturing Instructions used as the basis for the FMEA are shown at Annex A. Details of the FMEA are detailed in Table 2.
- 3.5. A list of definitions for the severity [S], occurrence [O] and detectability [D] ratings employed in the FMEA are shown in Table 3.

## 4. DISCUSSION

### 4.1. Reactor Dump Mode

4.1.1. The most safety-critical failure mode response is the 'DUMP MODE'. This mode is designed to prevent a runaway reaction from becoming critical, by drenching the reaction with approximately 3,000 gallons of cold filtered water and cooling in a recrystallization still (i.e. DUMP TANK) under agitation.

4.1.2. There are two scenarios that can cause a DUMP MODE to be initiated:

4.1.2.1. An automatic response to a 'HI-HI' temperature alarm within the reactor (configured for temperatures  $>99.5^{\circ}\text{C}$  at the time of writing).

4.1.2.2. Manual selection of 'DUMP' using the software interface by the operator at any time during a reaction run.

4.1.3. The DUMP MODE is designed to do the following:

4.1.3.1. Switch off the explosive feed (if in operation).

4.1.3.2. Switch on the agitator (if not already running).

4.1.3.3. Switch on the recirculation pump (if not already running).

4.1.3.4. Switch on the discharge pump.

4.1.3.5. Initiate full cooling to the reactor.

4.1.3.6. Position the discharge valve at the bottom of the reactor to direct the reactor contents to the DUMP TANK line.

4.1.3.7. Open the filtered water valve to flood the reactor with 2,000 gallons of cold filtered water.

4.1.3.8. Switch on the agitator in the DUMP TANK. (The DUMP TANK cooling water is switched on as part of the pre-operation startup activities, to ensure the DUMP TANK contents are thoroughly cooled).

4.1.4. The DUMP MODE effectively floods the reactor with 2,000 gallons of cold water whilst simultaneously pumping the reactor contents to the DUMP TANK (which will contain 1,000 gallons of cold water). This will rapidly remove any heat source, thus slowing down any exothermic reaction and bring a runaway reaction under control.

4.1.5. During a DUMP MODE, the reactor is emptied by typically two and up to three fully independent ways:

- 4.1.5.1. The recirculation pump is capable of pumping the reactor contents to the DUMP TANK.
- 4.1.5.2. The discharge pump and associated piping (which does not use any of the recirculation piping) is capable of pumping the reactor contents to the DUMP TANK.
- 4.1.5.3. Finally, in the event of either or both of the recirculation and discharge pumping processes failing then the reactor is designed to overflow the reactor contents into an overflow line, which is connected to the DUMP TANK using fully independent piping (4" diameter). This overflow line allows the DUMP TANK to be filled from the reactor by gravity alone, and is thus independent of power to the building.
- 4.1.6. At the end of the DUMP MODE, the agitator, pumps and filtered water automatically switch off. The DUMP TANK will contain approximately 5,000 gallons of liquid (mostly cold water), which will need to be re-processed in the reactor in order to complete the hydrolysis reaction.
- 4.1.7. The DUMP MODE has been tested in the HYDROLYSIS facility as part of the inert commissioning trials. The DUMP MODE performs satisfactorily and in accordance with the original process design criteria.

## **4.2. Other Failure Modes**

- 4.2.1. In addition to the safety-critical DUMP MODE, a number of other failure modes, of lesser importance (safety perspective); have been evaluated by creating deliberate equipment / process failures in the reactor (see Table 1). The failure modes were created by either providing false signals to the PLC and/or by removal of component fuzes and/or by switching valves deliberately to incorrect positions.
- 4.2.2. During these trials, no failure modes have been identified which resulted in conditions that could lead to an unplanned or uncontrolled incident that could cause harm to personnel or significant damage to the facility. The equipment behaves as designed and performs satisfactorily as far as can be determined at this time.

## **4.3. FMEA**

- 4.3.1. The FMEA conducted on the HYDROLYSIS process pre-operational checks Manufacturing Instructions highlighted a number of potential risks. Most of the identified risks are considered acceptable and are in-line with the previously reported FMEA. Suggested preventative action has been recommended for some of the Manufacturing Instructions. It is recommended that these suggested improvements be implemented prior to using the facility for routine production using RONA Process Operators.

## **5. CONCLUSIONS AND RECOMMENDATIONS**

- 5.1. Failure modes for the HYDROLYSIS facilities located in HSAAP Building G-10 have been evaluated using the equipment. A number of theoretical process and equipment faults have been artificially created. The impact of these failure modes has been evaluated and has been found to be satisfactory.
- 5.2. The safety critical reactor DUMP MODE has been thoroughly tested under a number of conditions, some of which have involved multiple theoretical facility and/or process failures. The DUMP MODE has performed in accordance with the design expectations for the facility and performs satisfactorily.
- 5.3. A Failure Modes and Effects Analysis (FMEA) has been conducted on the HYDROLYSIS Manufacturing Instructions. The FMEA has identified a number of process and facility risks, which are in-line with a previous, detailed FMEA study. A number of additional suggested improvements have been identified, the implementation of which are recommended prior to using the facility for routine production using RONA Operations staff.
- 5.4. In terms of process and equipment failure modes, the HYDROLYSIS FACILITY is now ready for the commencement of live commissioning trials.
- 5.5. It is recommended that this failure modes evaluation report be reviewed and if necessary updated following the completion of live commissioning trials and prior to formally handing the facility to RONA Operations Department.

## **6. REFERENCES**

- 1 - FMEA Team Report 'FAILURE MODES AND EFFECTS ANALYSIS (FMEA) OF THE EXPLOSIVES HYDROLYSIS PROCESS AT THE HOLSTON ARMY AMMUNITION PLANT (HSAAP)', issued November 2000.

## **7. DISTRIBUTION**

- 7.1. Mr. N.House, RONA Operations Director.
- 7.2. Mr.A.Wilson, RONA R&D Director.
- 7.3. Mr. G.Plum, RONA Safety Manager.
- 7.4. Mr. J.Hammonds, RONA Explosives Division Manager.
- 7.5. Mr. L.Barnett, RONA Operations Process Engineer.
- 7.6. Mr. M.Ervin, RONA R&D Scientist.
- 7.7. Mr. Steve Taylor, RONA Environmental Engineer.
- 7.8. Mr. A.Lemke, HSAAP Government Safety Advisor.

**TABLE 1 – FAILURE MODES EVALUATED ON THE HYDROLYSIS FACILITY AT HSAAP BUILDING G-10**

	<b>FAILURE MODE EVALUATED</b>	<b>EQUIPMENT RESPONSE</b>	<b>COMMENT / RECOMMENDATION</b>
1	Interlocks on valves HV-19-1, HV-12-1, HV-41 (valves left in wrong positions deliberately)	Cannot initiate a batch unless valves are in correct position.	Work as designed.
2	Recirculation pump failure (pump fuze removed during operation)	Shuts off energetic feeder (if active). No other action taken	Works as designed. An alarm signal to inform Operator of pump failure would aid operability.
3	Agitator failure (fuze removed during operation)	Shuts off energetic feeder (if active). No other action taken	Works as designed. An alarm signal to inform Operator of pump failure would aid operability.
4	Over temperature (HI; >90°C)	Full cooling applied to the reactor and all feeds shut off automatically.	Works as designed.
5	Over temperature (HI-HI; >99.5°C)	Full cooling applied to reactor, all feeds shut off, recirculation pump started, discharge valve opened, discharge pump started, 2,000 gallons of cold filtered water fed into reactor at maximum flow rate – all occur automatically with no manual intervention. Reactor contents pumped to ‘DUMP TANK’ (referred to as DUMP MODE hereafter). At the end of a DUMP MODE, the agitator and pumps are switched off.	Works as designed.
6	Manual ‘DUMP’ mode	Invokes a DUMP MODE per the above.	Works as designed.
7	Manual ‘DUMP’ mode with	Invokes a DUMP MODE per the above	Reactor dumps as designed, except that the level in the reactor increases whilst

	<b>FAILURE MODE EVALUATED</b>	<b>EQUIPMENT RESPONSE</b>	<b>COMMENT / RECOMMENDATION</b>
	recirculation pump disabled (fuze removed)		water is added. This can cause the reactor contents to leak thro' the reactor agitator water seal. Contents overflow into dump tank using overflow line (per design). Recommendation is to add a second reactor seal to prevent such a leak.
8	Manual 'DUMP' mode with discharge pump disabled (fuze removed)	Invokes a DUMP MODE per the above.	Behaves as for (7), except that the reactor level rises more slowly.
9	Manual 'DUMP' mode with discharge pump and recirculation pump disabled (fuzes removed)	Invokes a DUMP MODE per the above.	Behaves as for 7 except that the reactor level rises more quickly. Significant overflow of reactor contents into diked area otherwise proceeds very well. Additional reactor sealing (see 7) will improve the performance of the equipment.
10	Manual DUMP during explosive feed cycle.	Invokes DUMP MODE per the above	Works as designed.
11	Manual DUMP during digest reaction cycle.	Invokes DUMP MODE per the above.	Works as designed.
12	Reactor level high (HI alarm)	Shuts off all feeds.	Works as designed. Note: interlock overridden during DUMP MODE cycle – reactor will continue a DUMP MODE irrespective of reactor level.
13	Flame detector	Floods Feeder hopper with water. Water directed straight	Works as designed.

	<b>FAILURE MODE EVALUATED</b>	<b>EQUIPMENT RESPONSE</b>	<b>COMMENT / RECOMMENDATION</b>
	above Loss in Weight Feeder activated using a naked flame source	into feed hopper. Water flow will continue until manually shut off.	
14	Temperature probe failure (T1)	PLC automatically determines an accurate temperature from either of the two temperature probes. The PLC uses the higher of the two values as the actual temperature of the reactor contents.	Works as designed.
15	Building G-10 Power Failure		



**TABLE 2 – RESULTS OF AN FMEA ON THE HYDROLYSIS FACILITY MANUFACTURING INSTRUCTIONS**

<b>PROCESS: HYDROLYSIS OF ENERGETIC MATERIALS (Manufacturing Instructions)</b>					<b>ASSESSORS: A.Wilson, G.Plum, A.Lemke.</b>					<b>ESTIMATED IMPACT</b>			
<b>Ref.</b>	<b>POTENTIAL FAILURE MODE</b>	<b>POTENTIAL EFFECTS</b>	<b>POTENTIAL CAUSES</b>	<b>[S]</b>	<b>CURRENT CONTROLS</b>	<b>[O]</b>	<b>[D]</b>	<b>RPN</b>	<b>RECOMMENDED ACTION</b>	<b>[S]</b>	<b>[O]</b>	<b>[D]</b>	<b>RPN</b>
<b>2 PRE-OPERATIONAL CHECKLISTS</b>													
<b>2.1 PRE-OPERATIONAL CHECKLIST FOR G-10 TANK FARM</b>													
2.1.1	Hydrolysate discharge valves HV-10, HV-12, HV-14 or HV-15 open or partially open on tanks GT-10, GT12, GT14 or GT-15	Hydrolysate lines fill with hydrolysate, potentially mixing hydrolysate types subsequently requiring off-site disposal.	Human error.	5	MI and SOP	3	8	120	None	5	3	8	120
2.1.2	Caustic valve HV-21-1 closed or partially closed	No / low flow of caustic into reactor during subsequent process. Minor program delays.	Human error.	3	MI and SOP	5	3	45	None	3	5	3	45
			Mechanical failure of valve (leaking)	3	Preventative Maintenance	5	3	45	Transfer caustic to dump tank pending repair of leaking valve [C]	3	5	3	45

PROCESS: HYDROLYSIS OF ENERGETIC MATERIALS (Manufacturing Instructions)					ASSESSORS: A.Wilson, G.Plum, A.Lemke.					ESTIMATED IMPACT			
Ref.	POTENTIAL FAILURE MODE	POTENTIAL EFFECTS	POTENTIAL CAUSES	[S]	CURRENT CONTROLS	[O]	[D]	RPN	RECOMMENDED ACTION	[S]	[O]	[D]	RPN
2.1.3	Acid discharge valve HV-22-1 open or partially open	Acid leaks into acid line. Potential for acid entering reactor out with process procedure (requires additional failure), which would ruin experiment.	Human error.	5	PLC checks status of acid valve prior to operation. MI and SOP	5	1	25	None	5	5	1	25
2.1.4	Dike drain valves open or partially open.	If tank(s) developed leak(s) then contents would flow to Waste Water Treatment Facilities, possibly violating permit incurring financial penalties.	Human error.	8	MI and SOP	3	8	192	None	8	3	8	192

PROCESS: HYDROLYSIS OF ENERGETIC MATERIALS (Manufacturing Instructions)					ASSESSORS: A.Wilson, G.Plum, A.Lemke.					ESTIMATED IMPACT			
Ref.	POTENTIAL FAILURE MODE	POTENTIAL EFFECTS	POTENTIAL CAUSES	[S]	CURRENT CONTROLS	[O]	[D]	RPN	RECOMMENDED ACTION	[S]	[O]	[D]	RPN
2.1.5	Water / condensate not drained from Diked area.	Difficult to visually determine tank integrity (I.e. for leaks) from a casual inspection.	Human error.	1	MI and SOP	8	6	48	None	1	8	6	48
<b>2.2 PRE-OPERATIONAL CHECKLIST AND PREPARATION OF REMOTE SAMPLER INSIDE BUILDING G-10</b>													
2.2.1	Valve FV-1 closed or partially closed.	No / reduced flow to Feeder in the event of a fire being detected by flame eye, resulting in detonation (requires second failure).	Human error.	9	MI and SOP	5	6	270	Fit pressure sensor to deluge line and link to PLC.	9	2	3	54
2.2.2.1	Sampler drain valve open or partially open.	Water leaks from sampler during operation, possibly preventing samples to remain chilled.	Human error.	3	MI and SOP	5	3	45	None	3	5	3	45

PROCESS: HYDROLYSIS OF ENERGETIC MATERIALS (Manufacturing Instructions)					ASSESSORS: A.Wilson, G.Plum, A.Lemke.					ESTIMATED IMPACT			
Ref.	POTENTIAL FAILURE MODE	POTENTIAL EFFECTS	POTENTIAL CAUSES	[S]	CURRENT CONTROLS	[O]	[D]	RPN	RECOMMENDED ACTION	[S]	[O]	[D]	RPN
			Mechanical failure of valve (leaking)	3	Preventative Maintenance	3	3	27	Repair valve and continue with process [C].	3	5	3	45
2.2.2.3	Insufficient ice loaded into bin	Samples not chilled adequately.	Human error.	5	MI and SOP	5	6	150	None	5	5	6	150
2.2.2.5	Insufficient ice loaded into center compartment	Samples not chilled adequately.	Human error.	5	MI and SOP	5	6	150	None	5	5	6	150
2.2.2.6	Too little / no water	Samples not chilled adequately.	Human error.	5	MI and SOP	5	6	150	None	5	5	6	150
2.2.2.7	Bottles installed in wrong order	Potentially void experimental results.	Human error.	2	MI and SOP	5	8	80	Clearly label bottles. MI to specify final check of bottle configuration.	2	3	3	18
	Bottles secured incorrectly - fall off before or during experiment.	Potentially hazardous ice-bath contents. Ruined sample(s).	Human error.	6	MI and SOP	9	8	432	MI to specify final check to ensure sample bottles are secured correctly.	6	8	3	144
	No quenching heel in sample	High pH of final sample, causing	Human error.	3	MI and SOP	3	3	27	None	3	3	3	27

PROCESS: HYDROLYSIS OF ENERGETIC MATERIALS (Manufacturing Instructions)					ASSESSORS: A.Wilson, G.Plum, A.Lemke.					ESTIMATED IMPACT			
Ref.	POTENTIAL FAILURE MODE	POTENTIAL EFFECTS	POTENTIAL CAUSES	[S]	CURRENT CONTROLS	[O]	[D]	RPN	RECOMMENDED ACTION	[S]	[O]	[D]	RPN
	bottle.	invalid results.											
	Too much quenching heel in sample bottle.	Invalid testing results.	Human error.	3	MI and SOP	3	3	27	None	3	3	3	27
	Incorrect quenching chemical added to sample bottle.	Incompatibility, possible gassing / reaction leading to personnel exposure.	Human error.	8	Trained Laboratory technicians, familiar with sample preparation.	2	8	128	None	8	2	8	128
2.2.2.8	Water batch not cranked up high enough.	Samples not chilled adequately.	Human error.	5	MI and SOP	5	6	150	None	5	5	6	150
2.2.3	Valves 201-A and 201-B closed or partially closed.	Vapors enter scrubber. Concentrations greater than expected.	Human error.	3	Scrubber capable of coping with significant effluent. MI and SOP.	5	8	120	None	3	5	8	120
2.2.4	Valve A-1 closed or partially open	Reduced or no acid flow during neutralization process step.	Human error.	1	Neutralization stage delayed until valve opened. No significant issue.	5	3	15	None	1	5	3	15

PROCESS: HYDROLYSIS OF ENERGETIC MATERIALS (Manufacturing Instructions)					ASSESSORS: A.Wilson, G.Plum, A.Lemke.					ESTIMATED IMPACT			
Ref.	POTENTIAL FAILURE MODE	POTENTIAL EFFECTS	POTENTIAL CAUSES	[S]	CURRENT CONTROLS	[O]	[D]	RPN	RECOMMENDED ACTION	[S]	[O]	[D]	RPN
2.2.5	Valve C-1 closed or partially open	Reduced or no caustic flow during process. Reaction will not proceed.	Human error.	5	Caustic flow, pH monitored by PLC. Explosive feed will not be permitted by PLC. No significant issue.	3	6	90	None	5	3	6	90
2.2.6	Air sample valve 100 not open or partially open	Poor gas samples, ruining experiment	Human error.	3	MI and SOP	5	6	90	None	3	5	6	90
2.2.7	Valve 23-1 open or partially open	Dump tank contents can be pumped to reactor (requires a second failure).	Human error.	3	Reactor contents would overflow back into dump tank.	2	6	36	None	3	2	6	36
2.2.8	Valve AS-1 Closed or partially open.	Reactor contents seep into agitator seal, leaving an energetic residue, which ignites at a later time.	Human error.	8	MI and SOP	5	6	240	MI to specify check for water discharge on north side of reactor, confirming flow.	8	5	3	120

PROCESS: HYDROLYSIS OF ENERGETIC MATERIALS (Manufacturing Instructions)					ASSESSORS: A.Wilson, G.Plum, A.Lemke.					ESTIMATED IMPACT			
Ref.	POTENTIAL FAILURE MODE	POTENTIAL EFFECTS	POTENTIAL CAUSES	[S]	CURRENT CONTROLS	[O]	[D]	RPN	RECOMMENDED ACTION	[S]	[O]	[D]	RPN
2.2.9	Valve R-1 Closed or partially open.	No / reduced recirculation of reactor contents during operation.	Human error.	2	PLC detects flow in recirculation line and initiates auto-shut down.	3	3	18	None	2	3	3	18
2.2.10	Valve 201-C closed or partially open.	No reactor cooling, leading to heat build up in reactor and reaction runaway.	Human error.	2	PLC detects and informs operator of temperatures. MI and SOP. Auto dumping of reactor in event of over temperature.	5	6	60	None	2	5	6	60
2.2.11	Valve ST-1 on Sample Line closed or partially open.	Tracer line not heated, resulted in incorrect samples taken during experiment.	Human error.	3	MI and SOP	5	8	120	None	3	5	8	120
2.2.12	Valve ST-1 supplying steam to heat exchanger closed or partially open.	Heat exchanger unable to heat reactor contents during operation. Experiment delayed.	Human error.	1	PLC checks reactor temperature during operation.	8	3	24	None	1	8	3	24

PROCESS: HYDROLYSIS OF ENERGETIC MATERIALS (Manufacturing Instructions)					ASSESSORS: A.Wilson, G.Plum, A.Lemke.					ESTIMATED IMPACT			
Ref.	POTENTIAL FAILURE MODE	POTENTIAL EFFECTS	POTENTIAL CAUSES	[S]	CURRENT CONTROLS	[O]	[D]	RPN	RECOMMENDED ACTION	[S]	[O]	[D]	RPN
2.2.13	Valve 201-D closed or partially open.	No reactor cooling, leading to heat build up in reactor and reaction runaway.	Human error.	2	PLC detects and informs operator of temperatures. MI and SOP. Auto dumping of reactor in event of over temperature.	5	6	60	None	2	5	6	60
2.2.14	Valve HV-7-1 open or partially open	Reactor contents leak during experiment, resulting in dump tank being partially filled with hydrolysate / caustic and failed experiment. Potential for personnel exposure.	Human error.	8	MI and SOP	5	8	320	Fit sensor to valve and link to PLC; program such that reactor cannot operate whilst valve is not fully closed.	8	5	1	40
2.2.15	Valve 19-1 open or partially open.	Would change reactor dumping cycle and may redirect material to tank farm.	Human error.	2	PLC checks and acts upon status of valve prior to operation of reactor.	3	3	18	None	2	3	3	18



PROCESS: HYDROLYSIS OF ENERGETIC MATERIALS (Manufacturing Instructions)					ASSESSORS: A.Wilson, G.Plum, A.Lemke.					ESTIMATED IMPACT			
Ref.	POTENTIAL FAILURE MODE	POTENTIAL EFFECTS	POTENTIAL CAUSES	[S]	CURRENT CONTROLS	[O]	[D]	RPN	RECOMMENDED ACTION	[S]	[O]	[D]	RPN
		Partial reactor contents would fill hydrolysate line to tank farm during dump cycle. Potential for partial, reduced-rate hydrolysis reaction in hydrolysate line (unheated, uninsulated line). Not considered a significant hazard.	Mechanical failure of valve (leaking)	6	Preventative Maintenance	5	8	240	None	6	5	8	240
	Valve 42 open or partially open.	Would change reactor dumping cycle and may redirect material to loading dock.	Human error.	2	PLC checks and acts upon status of valve prior to operation of reactor.	3	3	18	None	2	3	3	18

PROCESS: HYDROLYSIS OF ENERGETIC MATERIALS (Manufacturing Instructions)					ASSESSORS: A.Wilson, G.Plum, A.Lemke.					ESTIMATED IMPACT			
Ref.	POTENTIAL FAILURE MODE	POTENTIAL EFFECTS	POTENTIAL CAUSES	[S]	CURRENT CONTROLS	[O]	[D]	RPN	RECOMMENDED ACTION	[S]	[O]	[D]	RPN
		Partial reactor contents would fill hydrolysate line to loading dock during dump cycle. Potential for partial, reduced-rate hydrolysis reaction in hydrolysate line (boxway heated, uninsulated line). Not considered a significant hazard.	Mechanical failure of valve (leaking)	6	Preventative Maintenance	5	8	240	None	6	5	8	240
	Valve HV-12-2 open or partially open	No impact on hydrolysis process, would delay hydrolysis line cleaning only.	Human error.	2	MI and SOP	5	6	60	None	2	5	6	60

PROCESS: HYDROLYSIS OF ENERGETIC MATERIALS (Manufacturing Instructions)					ASSESSORS: A.Wilson, G.Plum, A.Lemke.					ESTIMATED IMPACT			
Ref.	POTENTIAL FAILURE MODE	POTENTIAL EFFECTS	POTENTIAL CAUSES	[S]	CURRENT CONTROLS	[O]	[D]	RPN	RECOMMENDED ACTION	[S]	[O]	[D]	RPN
	Valve 12-1 closed or partially open.	Would prevent reactor dumping via bottom of reactor valve.	Human error.	2	PLC checks and acts upon status of valve prior to operation of reactor. Reactor has two other independent means of emptying into dump tank.	3	3	18	None	2	3	3	18
2.2.16	Valve ST-3 closed or partially open.	No / reduced steam heating of gas sample line, leading to incorrect gas sample.	Human error.	3	MI and SOP	5	8	120	None	3	5	8	120
2.2.17	Dump tank filled with too much water	Dump tank contents would overflow to catch-basin.	Human error.	5	Dump tank level indicator linked to PLC. Reactor will not start unless tank only partially filled. MI and SOP	5	6	150	None	5	2	3	30
	Dump tank filled with too little water	Reactor contents would not be adequately quenched, possible	Human error.	9	Dump tank level indicator linked to PLC. Reactor will not start unless tank only partially filled. MI and SOP	5	6	270	None	9	2	3	54

PROCESS: HYDROLYSIS OF ENERGETIC MATERIALS (Manufacturing Instructions)					ASSESSORS: A.Wilson, G.Plum, A.Lemke.					ESTIMATED IMPACT			
Ref.	POTENTIAL FAILURE MODE	POTENTIAL EFFECTS	POTENTIAL CAUSES	[S]	CURRENT CONTROLS	[O]	[D]	RPN	RECOMMENDED ACTION	[S]	[O]	[D]	RPN
		runaway reaction in dump tank.											
	Dump tank filled with liquid other than water (e.g. hydrolysate)	No significant impact - hydrolysate solution essentially as capable of quenching reaction as water.	Human error.	2	MI and SOP	3	8	48	None	2	3	8	48
2.2.18	Valve AS-2 closed or partially open.	Dump tank contents seep into dump tank seal, leading to an ignition at a later time.	Human error.	1	PLC checks water flow - auto shut down if flow drops below a pre-defined minimum. MI and SOP.	3	6	18	None	1	3	6	18
2.2.19	Scrubber flow water valve SW-1 closed or partially open.	Reactor gas emissions not scrubbed. Potential for gas emissions	Human error.	5	PLC checks for water flow and signals if low. MI and SOP.	5	3	75	None	5	5	3	75

PROCESS: HYDROLYSIS OF ENERGETIC MATERIALS (Manufacturing Instructions)					ASSESSORS: A.Wilson, G.Plum, A.Lemke.					ESTIMATED IMPACT			
Ref.	POTENTIAL FAILURE MODE	POTENTIAL EFFECTS	POTENTIAL CAUSES	[S]	CURRENT CONTROLS	[O]	[D]	RPN	RECOMMENDED ACTION	[S]	[O]	[D]	RPN
2.2.20	Valve 41 closed or partially open.	Reduced / no overflow capability from reactor to dump tank. Potential for reactor flooding thro' scrubber during dump cycle (requires at least one other failure).	Human error.	5	PLC checks that valve is open. Reactor cannot start unless valve position is correct. MI and SOP	5	3	75	None	5	5	3	75
2.2.21	Valve 10-3 off or partially on.	Reduced reactor cooling capability, leading to over temperature during process.	Human error.	3	Reactor dump cycle and thermal inertia of reactor. Not expected to result in any major safety issues.	5	8	120	None	3	5	8	120
2.2.22	Valve HS 10-2 in incorrect position.	Potential for heating of dump tank, reducing the effectiveness of	Human error.	1	PLC detects dump tank temperature. MI and SOP	5	6	30	PLC to flag an alarm if dump tank temperature exceeds 40°C	1	5	3	15

PROCESS: HYDROLYSIS OF ENERGETIC MATERIALS (Manufacturing Instructions)					ASSESSORS: A.Wilson, G.Plum, A.Lemke.					ESTIMATED IMPACT			
Ref.	POTENTIAL FAILURE MODE	POTENTIAL EFFECTS	POTENTIAL CAUSES	[S]	CURRENT CONTROLS	[O]	[D]	RPN	RECOMMENDED ACTION	[S]	[O]	[D]	RPN
		the dump mode, but not the dump capability.											
2.2.23	Valve D-2 open or partially open.	Dyked area would not automatically drain to dump tank in the event of a spillage.	Human error.	1	MI and SOP	3	2	6	None	1	3	2	6
2.2.24	Valve D-1 open or partially open	Dump tank contents would leak into hydrolysate lines. Potential for hydrolysates to mix.	Human error.	5	MI and SOP	8	6	240	None	5	8	6	240
2.2.25	Valve C-2 closed or partially closed.	Unable to load caustic to tank farm	Human error.	1	MI and SOP	5	3	15	None	1	5	3	15
	Valve C-3 closed or partially closed.	Unable to load caustic into reactor.	Human error.	1	MI and SOP	5	3	15	None	1	5	3	15
	Valve C-4 closed or partially closed.	Unable to load caustic into reactor.	Human error.	1	MI and SOP	5	3	15	None	1	5	3	15

**TABLE 3 – DEFINITIONS FOR THE FMEA SEVERITY, OCCURRENCE  
AND DETECTABILITY VALUES**

No.	DEFINITION		
	SEVERITY [S]	OCCURRENCE [O]	DETECTABILITY [D]
1	No significant impact (safety, cost or timescale).	Never been known to occur anywhere.	Obvious; cannot easily be missed by staff likely to be present.
2	Some administration issues, minor cost impact.	Never been known to occur at HSAAP based on past performance; considered unlikely to occur in process.	Not used
3	Minor schedule impact	Similar occurrence known to have occurred at least once at HSAAP in the past, but considered rare.	Detectable by a casual inspection only
4	Not used	Not used	Not used
5	Program delays expected. Moderate cost impact likely.	Occurred more than once in the past at HSAAP in a similar activity – a realistic but probably unlikely occurrence for the process.	Not used
6	Expect equipment damage. Potential for minor personnel exposure	Not used	Detectable as part of the standard process inspection / monitoring only.
7	Major cost and/or schedule delay; no significant personnel safety issues	Not used	Not used
8	Expect minor personnel health risks and/or significant equipment damage / cost / delay	Expected to occur at least once during the planned experimental trials for the program.	Detectable only if specifically looked for; will probably not otherwise be detected.
9	Imminent danger to personnel and/or substantial equipment / facility damage and/or cost/delays. Very unsafe.	Expected to occur more than once during the planned experimental trials for the program.	Unlikely to find without an intensive specific search.
10	Catastrophic event to personnel and/or damage to facility. Prohibitive cost / schedule impact likely	Frequent occurrence during the life of the program expected.	Probably undetectable until too late; cannot reasonably be expected to be found

## FMEA – MANUFACTURING INSTRUCTIONS

1. The following text relates to the proposed procedure for operating the HYDROLYSIS FACILITY. The instructions have been evaluated in the Failure Modes and Effects Analysis (FMEA) reported herein. The instructions have been incorporated into the HYDROLYSIS FACILITY Manufacturing Instructions as part of the overall facility documentation.

### **2. PRE-OPERATIONAL CHECKLISTS**

#### **2.1 Pre-operational Checklist Prior to Operating the Reactor - Building G-10 Tank Farm**

- 2.1.1** Manually close the valves (identified as HV-10, HV-12, HV-14, and HV-15) on the discharge lines of Tank Nos. GT-10, GT-12, GT-14, and GT-15 that are designated for storage of the hydrolysate product.
- 2.1.2** Manually open the valve (identified as No. HV-21-1) located on the discharge line of the GT-11 Caustic Storage Tank.
- 2.1.3** Manually close (or confirm closed) the valve (identified as HV-22-1) located on the discharge line of the GT-13 Acid Storage Tank.
- 2.1.4** Manually close the two dike drain valves located on the north side of the two diked areas in the Building G-10 Tank Farm using a tee-handle.
- 2.1.5** If upon visual inspection, liquid is visible inside the diked area then inform supervision who will determine whether the liquid is the result of a leaking tank or simply water from rainfall or steam condensate. If any observed liquid is determined to be simply rainfall or steam condensate then open the two-dike drain valves to drain water to the industrial sewer. Close the valves immediately upon completion of this operation.

#### **2.2 Pre-operational Checklist Prior to Operating the Reactor - Inside Building G-10**

- 2.2.1** Manually open (or confirm open) the valve (identified as No. FV-1 and located on the third floor {west side} of Building G-10 adjacent to the material lift bay) which supplies water to the system deluge nozzle above the feed hopper.
- 2.2.2** Isolate the sampler to prevent remote operation by switching the SAMPLER E-STOP lever to the off position. The E-STOP lever is located on the west wall adjacent to the sampler in Building G-10. The remote sampling cabinet can now be prepared as follows:
  - 2.2.2.1** Close the drain valve located on the ice water bin in the sample cabinet.
  - 2.2.2.2** Remove the screen from the sample tray in the sample cabinet.
  - 2.2.2.3** Load the sample bin to the top of the screen support with ice, and replace the screen.
  - 2.2.2.4** Install the four latches to secure the screen.
  - 2.2.2.5** Place ice in the center compartment on top of the screen.



- 2.2.2.6** Using the water hose mounted on Column C-3 in the west side of Building G-10, manually add water to the sample bin filled with ice until the level reaches indicator mark approximately one inch from the top lip of the ice bin.
- 2.2.2.7** Load the new sample bottles, containing the pre-measured acid heel for quenching, into the sample carousel. (Make sure all bottles are threaded snugly into the teflon bottle holders.)
- 2.2.2.8** Using the manual crank, lift the water bath to its maximum height, this is at an appropriate level to partially cover the sample bottles in the ice water.
- 2.2.2.9** Close the door(s) of the sample cabinet and secure with the latches.
- 2.2.2.10** Switch the SAMPLER E-STOP lever to the ON position to activate the sampler thereby enabling computer control.
- 2.2.3** Manually open the two valves (identified as Nos. 201-A and 201-B) on the line providing cooling water to the condenser located on the north side of the reactor skid (upper level) opposite of the manhole cover.
- 2.2.4** Manually open the valve (identified as No. A-1) installed in series with the automated valve on the acid feed line located on the east side of the reactor skid (upper level).
- 2.2.5** Manually open the valve (identified as No. C-1) installed in series with the automated valve on the caustic feed line located on the east side of the reactor skid (upper level).
- 2.2.6** Manually open the air sample valve (identified as No. 100) on the port on the gas sampling valve located on the east side of the reactor skid (upper level).
- 2.2.7** Manually close the valve (identified as No. 23-1) on the pipe allowing dump tank contents to be pumped back to the reactor (located on the east side of the reactor skid (upper level) adjacent to the manhole cover).
- 2.2.8** Manually open the valve (identified as AS-1) providing water to the water seal of the agitator located on the south side of the reactor skid (upper level) next to the manhole cover. The flow of water should be adjusted to achieve maximum flow without overflowing the seal. Open the manhole cover and visually confirm that water is not leaking into the reactor. Also, check the discharge pipe (labeled as Agitator Seal Water) at the bottom of the reactor skid (north side) to ensure water is flowing.
- 2.2.9** Manually open the valve (identified as No. R-1) on the circulation loop of the reactor that is located adjacent to the manhole on the reactor.
- 2.2.10** Manually open the valve (identified as No. 201-C) on the line providing cooling water to the heat exchanger located on the north side of the reactor skid (lower level) opposite of the manhole cover.
- 2.2.11** Manually open (or confirm opened) the valve (identified as No. ST-1) supplying steam to the tracer line on Sample Valve No. 100 located on the north side of the reactor skid (lower level).
- 2.2.12** Manually open the valve (identified as ST-1) on the line supply steam to the reactor heat exchanger located on the north side of the reactor skid (lower level).

- 2.2.13** Manually open the valve (identified as No. 201-D) on the line allowing flow of water from the jacket on the reactor to the industrial sewer located on the west side of the reactor skid (lower level).
- 2.2.14** Manually close (or confirm closed) the valve (identified as No. HV-7-1) on the recirculation pump suction line, located on the south side of the reactor skid at ground level. (This valve will later be used to collect the final sample of hydrolysate product at the end of a reactor run.)
- 2.2.15** On the discharge side of the hydrolysate transfer pump (No. PU4), manually close Valve Nos. 19-1, HV-42, and HV-12-2. Also, at this position, manually open Valve No. HV-12-1 on the overflow line between the reactor and the dump tank.
- 2.2.16** Manually open the valve (identified as No. ST-3), located adjacent to Valve No. 19-1 at floor level, that supplies steam to the steam tracer line on the gas sampling line coming from the reactor.
- 2.2.17** Inspect the water level in the dump tank. If necessary, adjust the level to the top of the interior heating / cooling coils (approximately 2000 gallons).
- 2.2.17.1** If water needs to be added, open the valve (identified as No. W-1) on filter water inlet line to the dump tank (located on the west side of the dump tank adjacent to the manhole).
- 2.2.18** Manually open the valve (identified as No. AS-2) providing water to the water seal on the dump tank (located on the west side of the top of the dump tank). Visually confirm water flow by inspecting the drain line outlet in the drain at the north wall at the back of the dump tank.
- 2.2.19** Manually open the scrubber water valve (identified as No. SW-1) and set the water flow at 40 gallon per minute (gpm)  $\pm$  5 gpm.
- 2.2.20** Manually open the valve (identified as No. HV-41) located on the overflow line (first floor) between the reactor and the dump tank.
- 2.2.21** Turn on the river water flow valve (identified as 10-3) using hand-spring switch No. HS 10-3 (located on the first floor on the north side of the dump tank). The hand –spring switch No. HS 10-3 should be in the “Off” (“down”) position.
- 2.2.22** Turn off hand-switch valve No. HS 10-2 (i.e. switch in down position) located on the first floor on the south side of the dump tank. Valve No. 10-2 switches between steam-on the and water out of the dump tank heating / cooling coils.
- 2.2.23** Manually close (or confirm closed) the valve (identified as No. D-2), which allows liquid within the dyked area around the reactor to be drained to the dump tank, located on the south side of the dump tank (first floor).
- 2.2.24** Manually close (or confirmed closed) the valve (identified as No. D-1) located on the bottom of the dump tank that controls the flow of dump tank contents.
- 2.2.25** Manually close Valve No. C-2 and manually open Valve Nos. C-3 and C-4, which control the direction of flow in the caustic transfer lines, located on the first floor (west side) of Building G-10.

### **2.3 Pre-operational Checklist Prior to Operating the Reactor - In Building E-10 Control Room**

- 2.3.1** Note: the pre-operational activities in E-10 can be undertaken whilst Building G-10 is manned.
- 2.3.2** Switch on the computer and log in.
- 2.3.3** Adjust the remote feeder setpoints to feed the reactor with the desired quantity of energetic material (maximum 500 LB) at the desired feed rate as specified on the batch sheet.
- 2.3.4** Enter the desired reaction digest time into the PLC via the computer.
- 2.3.5** Click on the “START BATCH” icon in the computer screen to initiate a batch startup.
- 2.3.6** Check to ensure that the reactor tank level is less than 10% (which could mean zero) and that the measured temperature inside the reactor is less than 90°C. If either of these conditions are not true then consult with supervision.
- 2.3.7** Add water to the reactor (quantity as specified on the batch sheet).
- 2.3.8** Apply heating to the reactor, set point  $85\pm 2^{\circ}\text{C}$  unless otherwise specified on the batch sheet.

At this point, the reactor is ready for operation. Energetic materials are loaded into the loss in weight feeder, the building is checked and the reaction is conducted using the PLC. Full details are given in the Manufacturing Instruction.

## **APPENDIX J**

### **Failure Mode and Effects Analysis, Final**



ROYAL ORDNANCE NORTH AMERICA, Inc.

**HOLSTON ARMY AMMUNITION PLANT (HSAAP)**

**BUILDING G-10 ENERGETIC MATERIAL**

**HYDROLYSIS FACILITY FAILURE MODE**

**EVALUATION REPORT**

Compiled by:

*Signed*  
A.R.Wilson (R&D Director, RONA)

Date: 12 Dec 00

*Signed*  
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Date: 12 Dec 00

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Date: 12 Dec 00

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**CHANGE RECORD**

Issue Status	Affected pages
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A	Document issued to enable live G-10 facility commissioning
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Note: Amendments to this document will be achieved by re-issuing the entire document.

## **1. INTRODUCTION**

- 1.1. This document describes the failure modes evaluated in the new Holston Army Ammunition Plant Building G-10 Energetic Material Hydrolysis facility ('HYDROLYSIS FACILITY').
- 1.2. The failure modes were tested as part of the inert facility commissioning experiments during November / December 2000, a pre-requisite of live commissioning trials.
- 1.3. The failure modes were principally evaluated to verify the correct operation of the HYDROLYSIS FACILITY in the event of a given single failure mode.

## **2. HYDROLYSIS FACILITY**

- 2.1. A detailed description of the HYDROLYSIS FACILITY can be obtained by referring to the facility layout drawing (XYZ). A summary of the features of the HYDROLYSIS FACILITY are given below:
- 2.2. The nucleus of the HYDROLYSIS FACILITY consists of a 2,000 gallon glass-lined reactor (Pfaudler design), located in the center bay on the second floor of Building G-10. The reactor is fitted with comprehensive instrumentation:
  - 2.2.1. Volumetric flow meter devices fitted to all liquid feed ports (3).
  - 2.2.2. PH sensor (1).
  - 2.2.3. Redundant temperature probes in dip-tubes located in the reactor (2)
  - 2.2.4. Recirculation line with independent pump.
  - 2.2.5. Mass flow meter, fitted into the recirculation loop, with an additional option for indicating temperature or liquid density (1).
  - 2.2.6. Variable speed agitator.
  - 2.2.7. Pressurized heating / cooling thermocirculator (Temperature range nominally 30-100°C).
  - 2.2.8. Independent discharge pump for emptying the reactor.
- 2.3. The reactor can be fed solid feeds (energetic or inert) using an Acrison loss in weight feeder, located on the third floor of Building G-10 directly above the reactor. The feeder is designed to feed material at controlled feed rates up to 500 LB / hour. The feed hopper capacity is 500 LB (nominal).
- 2.4. The reactor is piped directly to an existing, previously unused Holston recrystallization still (nominal 6,000 gallon capacity). The still is located on the second floor, but a significant portion of the still volume is located under the second floor; the still is thus

“below” the glass lined reactor in terms of height. This enables the use of gravity flow between the reactor and the still, if required.

- 2.5. The still is designed to act as a highly capable secondary storage and processing tank for the contents of the reactor in the event of their being a reaction runaway or other major operational issue associated with the reactor. The still is termed ‘DUMP TANK’ for this reason, which term ‘DUMP TANK’ is used hereafter in this document. The contents of the reactor can be pumped or filled by gravity to the ‘DUMP TANK’ by three independent means (described in detail in a later section).
- 2.6. A remote sampling device is also fitted to the reactor to enable samples to be remotely taken from the reactor recirculation line during a reaction. This enables a reaction to be monitored. A maximum of sixteen (16) samples can be remotely obtained per experiment. The sample bottles can be pre-filled with a reaction quenching agent and/or flushed with quenching after being filled with a sample (remotely programmable). The samples can also be suspended in ice-cold water throughout an experiment (thereby aiding reaction quenching).

### **3. FAILURE MODE EVALUATION**

- 3.1. The failure modes evaluated relate to the critical operating parameters and failure modes identified in the conceptual Failure Mode and Effects Analysis conducted on the HYDROLYSIS FACILITY during CY 2000<sup>1</sup>.
- 3.2. The failure modes investigated and the results obtained are as shown in Table 1.
- 3.3. For each failure mode, an assessment has been made of the response of the equipment (PLC, software, reactor, feeder, dump-tank etc.). This assessment is summarized in Table 1 for each failure mode.
- 3.4. In addition to the failure mode experimental trials conducted on the HYDROLYSIS FACILITY, a Failure Modes and Effects Analysis (FMEA) has been conducted on the hydrolysis process pre-operational checks as defined in the Hydrolysis Process Manufacturing Instructions. The FMEA was conducted in accordance with the FMEA technique defined in the earlier FMEA report<sup>1</sup>. The Manufacturing Instructions used as the basis for the FMEA are shown at Annex A. Details of the FMEA are detailed in Table 2.
- 3.5. A list of definitions for the severity [S], occurrence [O] and detectability [D] ratings employed in the FMEA is shown in Table 3.



## 4. DISCUSSION

### 4.1. Reactor Dump Mode

- 4.1.1. The most safety-critical failure mode response is the 'DUMP MODE'. This mode is designed to prevent a runaway reaction from becoming critical, by drenching the reaction with approximately 3,000 gallons of cold filtered water and cooling in a recrystallization still (i.e. DUMP TANK) under agitation.
- 4.1.2. There are two scenarios that can cause a DUMP MODE to be initiated:
  - 4.1.2.1. An automatic response to a 'HI-HI' temperature alarm within the reactor (configured for temperatures  $>99.5^{\circ}\text{C}$  at the time of writing).
  - 4.1.2.2. Manual selection of 'DUMP' using the software interface by the operator at any time during a reaction run.
- 4.1.3. The DUMP MODE is designed to do the following:
  - 4.1.3.1. Switch off the explosive feed (if in operation).
  - 4.1.3.2. Switch on the agitator (if not already running).
  - 4.1.3.3. Switch on the recirculation pump (if not already running).
  - 4.1.3.4. Switch on the discharge pump.
  - 4.1.3.5. Initiate full cooling to the reactor.
  - 4.1.3.6. Position the discharge valve at the bottom of the reactor to direct the reactor contents to the DUMP TANK line.
  - 4.1.3.7. Open the filtered water valve to flood the reactor with 2,000 gallons of cold filtered water.
  - 4.1.3.8. Switch on the agitator in the DUMP TANK. (The DUMP TANK cooling water is switched on as part of the pre-operation startup activities, to ensure the DUMP TANK contents are thoroughly cooled).
- 4.1.4. The DUMP MODE effectively floods the reactor with 2,000 gallons of cold water whilst simultaneously pumping the reactor contents to the DUMP TANK (which will contain 1,000 gallons of cold water). This will rapidly remove any heat source, thus slowing down any exothermic reaction and bring a runaway reaction under control.
- 4.1.5. During a DUMP MODE, the reactor is emptied by typically two and up to three fully independent ways:

- 4.1.5.1. The recirculation pump is capable of pumping the reactor contents to the DUMP TANK.
- 4.1.5.2. The discharge pump and associated piping (which does not use any of the recirculation piping) is capable of pumping the reactor contents to the DUMP TANK.
- 4.1.5.3. Finally, in the event of either or both of the recirculation and discharge pumping processes failing then the reactor is designed to overflow the reactor contents into an overflow line, which is connected to the DUMP TANK using fully independent piping (4" diameter). This overflow line allows the DUMP TANK to be filled from the reactor by gravity alone, and is thus independent of power to the building.
- 4.1.6. At the end of the DUMP MODE, the agitator, pumps and filtered water automatically switch off. The DUMP TANK will contain approximately 5,000 gallons of liquid (mostly cold water), which will need to be re-processed in the reactor in order to complete the hydrolysis reaction.
- 4.1.7. The DUMP MODE has been tested in the HYDROLYSIS facility as part of the inert commissioning trials. The DUMP MODE performs satisfactorily and in accordance with the original process design criteria.

## **4.2. Other Failure Modes**

- 4.2.1. In addition to the safety-critical DUMP MODE, a number of other failure modes, of lesser importance (safety perspective), have been evaluated by creating deliberate equipment / process failures in the reactor (see Table 1). The failure modes were created by either providing false signals to the PLC and/or by removal of component fuzes and/or by switching valves deliberately to incorrect positions.
- 4.2.2. During these trials, no failure modes have been identified which resulted in conditions that could lead to an unplanned or uncontrolled incident that could cause harm to personnel or significant damage to the facility. The equipment behaves as designed and performs satisfactorily as far as can be determined at this time.

## **4.3. FMEA**

- 4.3.1. The FMEA conducted on the HYDROLYSIS process pre-operational checks Manufacturing Instructions highlighted a number of potential risks. Most of the identified risks are considered acceptable and are in-line with the previously reported FMEA. Suggested preventative action has been recommended for some of the Manufacturing Instructions. It is recommended that these suggested improvements be implemented prior to using the facility for routine production using RONA Process Operators.

## **5. CONCLUSIONS AND RECOMMENDATIONS**

- 5.1. Failure modes for the HYDROLYSIS facilities located in HSAAP Building G-10 have been evaluated using the equipment. A number of theoretical process and equipment faults have been artificially created. The impact of these failure modes has been evaluated and has been found to be satisfactory.
- 5.2. The safety critical reactor DUMP MODE has been thoroughly tested under a number of conditions, some of which have involved multiple theoretical facility and/or process failures. The DUMP MODE has performed in accordance with the design expectations for the facility and performs satisfactorily.
- 5.3. A Failure Modes and Effects Analysis (FMEA) has been conducted on the HYDROLYSIS Manufacturing Instructions. The FMEA has identified a number of process and facility risks, which are in-line with a previous, detailed FMEA study. A number of additional suggested improvements have been identified, the implementation of which are recommended prior to using the facility for routine production using RONA Operations staff.
- 5.4. In terms of process and equipment failure modes, the HYDROLYSIS FACILITY is now ready for the commencement of live commissioning trials.
- 5.5. It is recommended that this failure modes evaluation report be reviewed and if necessary updated following the completion of live commissioning trials and prior to formally handing the facility to RONA Operations Department.

## **6. REFERENCES**

- 1 - FMEA Team Report 'FAILURE MODES AND EFFECTS ANALYSIS (FMEA) OF THE EXPLOSIVES HYDROLYSIS PROCESS AT THE HOLSTON ARMY AMMUNITION PLANT (HSAAP)', issued November 2000.

## **7. DISTRIBUTION**

- 7.1. Mr. N.House, RONA Operations Director.
- 7.2. Mr.A.Wilson, RONA R&D Director.
- 7.3. Mr. G.Plum, RONA Safety Manager.
- 7.4. Mr. J.Hammonds, RONA Explosives Division Manager.
- 7.5. Mr. L.Barnett, RONA Operations Process Engineer.
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- 7.7. Mr. Steve Taylor, RONA Environmental Engineer.
- 7.8. Mr. A.Lemke, HSAAP Government Safety Advisor.

**TABLE 1 – FAILURE MODES EVALUATED ON THE HYDROLYSIS FACILITY AT HSAAP BUILDING G-10**

	<b>FAILURE MODE EVALUATED</b>	<b>EQUIPMENT RESPONSE</b>	<b>COMMENT / RECOMMENDATION</b>
1	Interlocks on valves HV-19-1, HV-12-1, HV-41 (valves left in wrong positions deliberately)	Cannot initiate a batch unless valves are in correct position.	Work as designed.
2	Recirculation pump failure (pump fuze removed during operation)	Shuts off energetic feeder (if active). No other action taken	Works as designed. An alarm signal to inform Operator of pump failure would aid operability.
3	Agitator failure (fuze removed during operation)	Shuts off energetic feeder (if active). No other action taken	Works as designed. An alarm signal to inform Operator of pump failure would aid operability.
4	Over temperature (HI; >90°C)	Full cooling applied to the reactor and all feeds shut off automatically.	Works as designed.
5	Over temperature (HI-HI; >99.5°C)	Full cooling applied to reactor, all feeds shut off, recirculation pump started, discharge valve opened, discharge pump started, 2,000 gallons of cold filtered water fed into reactor at maximum flow rate – all occur automatically with no manual intervention. Reactor contents pumped to ‘DUMP TANK’ (referred to as DUMP MODE hereafter). At the end of a DUMP MODE, the agitator and pumps are switched off.	Works as designed.
6	Manual ‘DUMP’ mode	Invokes a DUMP MODE per the above.	Works as designed.
7	Manual ‘DUMP’ mode with	Invokes a DUMP MODE per the above	Reactor dumps as designed, except that the level in the reactor increases whilst

	<b>FAILURE MODE EVALUATED</b>	<b>EQUIPMENT RESPONSE</b>	<b>COMMENT / RECOMMENDATION</b>
	recirculation pump disabled (fuze removed)		water is added. This can cause the reactor contents to leak thro' the reactor agitator water seal. Contents overflow into dump tank using overflow line (per design). Recommendation is to add a second reactor seal to prevent such a leak.
8	Manual 'DUMP' mode with discharge pump disabled (fuze removed)	Invokes a DUMP MODE per the above.	Behaves as for (7), except that the reactor level rises more slowly.
9	Manual 'DUMP' mode with discharge pump and recirculation pump disabled (fuzes removed)	Invokes a DUMP MODE per the above.	Behaves as for 7 except that the reactor level rises more quickly. Significant overflow of reactor contents into diked area, otherwise proceeds very well. Additional reactor sealing (see 7) will improve the performance of the equipment.
10	Manual DUMP during explosive feed cycle.	Invokes DUMP MODE per the above	Works as designed.
11	Manual DUMP during digest reaction cycle.	Invokes DUMP MODE per the above.	Works as designed.
12	Reactor level high (HI alarm)	Shuts off all feeds.	Works as designed. Note: interlock overridden during DUMP MODE cycle – reactor will continue a DUMP MODE irrespective of reactor level.
13	Flame detector	Floods Feeder hopper with water. Water directed straight	Works as designed.

	<b>FAILURE MODE EVALUATED</b>	<b>EQUIPMENT RESPONSE</b>	<b>COMMENT / RECOMMENDATION</b>
	above Loss in Weight Feeder activated using a naked flame source	into feed hopper. Water flow will continue until manually shut off.	
14	Temperature probe failure (T1)	PLC automatically determines an accurate temperature from either of the two temperature probes. The PLC uses the higher of the two values as the actual temperature of the reactor contents.	Works as designed.
15	Building G-10 Power Failure		

**TABLE 2 – RESULTS OF AN FMEA ON THE HYDROLYSIS FACILITY MANUFACTURING INSTRUCTIONS**

<b>PROCESS: HYDROLYSIS OF ENERGETIC MATERIALS (Manufacturing Instructions)</b>					<b>ASSESSORS: A.Wilson, G.Plum, A.Lemke.</b>					<b>ESTIMATED IMPACT</b>			
<b>Ref.</b>	<b>POTENTIAL FAILURE MODE</b>	<b>POTENTIAL EFFECTS</b>	<b>POTENTIAL CAUSES</b>	<b>[S]</b>	<b>CURRENT CONTROLS</b>	<b>[O]</b>	<b>[D]</b>	<b>RPN</b>	<b>RECOMMENDED ACTION</b>	<b>[S]</b>	<b>[O]</b>	<b>[D]</b>	<b>RPN</b>
<b>2 PRE-OPERATIONAL CHECKLISTS</b>													
<b>2.1 PRE-OPERATIONAL CHECKLIST FOR G-10 TANK FARM</b>													
2.1.1	Hydrolysate discharge valves HV-10, HV-12, HV-14 or HV-15 open or partially open on tanks GT-10, GT12, GT14 or GT-15	Hydrolysate lines fill with hydrolysate, potentially mixing hydrolysate types subsequently requiring off-site disposal.	Human error.	5	MI and SOP	3	8	120	None	5	3	8	120
2.1.2	Caustic valve HV-21-1 closed or partially closed	No / low flow of caustic into reactor during subsequent process. Minor program delays.	Human error.	3	MI and SOP	5	3	45	None	3	5	3	45
			Mechanical failure of valve (leaking)	3	Preventative Maintenance	5	3	45	Transfer caustic to dump tank pending repair of leaking valve [C]	3	5	3	45



PROCESS: HYDROLYSIS OF ENERGETIC MATERIALS (Manufacturing Instructions)					ASSESSORS: A.Wilson, G.Plum, A.Lemke.					ESTIMATED IMPACT			
Ref.	POTENTIAL FAILURE MODE	POTENTIAL EFFECTS	POTENTIAL CAUSES	[S]	CURRENT CONTROLS	[O]	[D]	RPN	RECOMMENDED ACTION	[S]	[O]	[D]	RPN
2.1.3	Acid discharge valve HV-22-1 open or partially open	Acid leaks into acid line. Potential for acid entering reactor outwith process procedure (requires additional failure), which would ruin experiment.	Human error.	5	PLC checks status of acid valve prior to operation. MI and SOP	5	1	25	None	5	5	1	25
2.1.4	Dike drain valves open or partially open.	If tank(s) developed leak(s) then contents would flow to Waste Water Treatment Facilities, possibly violating permit incurring financial penalties.	Human error.	8	MI and SOP	3	8	192	None	8	3	8	192

PROCESS: HYDROLYSIS OF ENERGETIC MATERIALS (Manufacturing Instructions)					ASSESSORS: A.Wilson, G.Plum, A.Lemke.					ESTIMATED IMPACT			
Ref.	POTENTIAL FAILURE MODE	POTENTIAL EFFECTS	POTENTIAL CAUSES	[S]	CURRENT CONTROLS	[O]	[D]	RPN	RECOMMENDED ACTION	[S]	[O]	[D]	RPN
2.1.5	Water / condensate not drained from Diked area.	Difficult to visually determine tank integrity (I.e. for leaks) from a casual inspection.	Human error.	1	MI and SOP	8	6	48	None	1	8	6	48
<b>2.2 PRE-OPERATIONAL CHECKLIST AND PREPARATION OF REMOTE SAMPLER INSIDE BUILDING G-10</b>													
2.2.1	Valve FV-1 closed or partially closed.	No / reduced flow to Feeder in the event of a fire being detected by flame eye, resulting in detonation (requires second failure).	Human error.	9	MI and SOP	5	6	270	Fit pressure sensor to deluge line and link to PLC.	9	2	3	54
2.2.2.1	Sampler drain valve open or partially open.	Water leaks from sampler during operation, possibly preventing samples to remain chilled.	Human error.	3	MI and SOP	5	3	45	None	3	5	3	45

PROCESS: HYDROLYSIS OF ENERGETIC MATERIALS (Manufacturing Instructions)					ASSESSORS: A.Wilson, G.Plum, A.Lemke.				ESTIMATED IMPACT				
Ref.	POTENTIAL FAILURE MODE	POTENTIAL EFFECTS	POTENTIAL CAUSES	[S]	CURRENT CONTROLS	[O]	[D]	RPN	RECOMMENDED ACTION	[S]	[O]	[D]	RPN
			Mechanical failure of valve (leaking)	3	Preventative Maintenance	3	3	27	Repair valve and continue with process [C].	3	5	3	45
2.2.2.3	Insufficient ice loaded into bin	Samples not chilled adequately.	Human error.	5	MI and SOP	5	6	150	None	5	5	6	150
2.2.2.5	Insufficient ice loaded into center compartment	Samples not chilled adequately.	Human error.	5	MI and SOP	5	6	150	None	5	5	6	150
2.2.2.6	Too little / no water	Samples not chilled adequately.	Human error.	5	MI and SOP	5	6	150	None	5	5	6	150
2.2.2.7	Bottles installed in wrong order	Potentially void experimental results.	Human error.	2	MI and SOP	5	8	80	Clearly label bottles. MI to specify final check of bottle configuration.	2	3	3	18
	Bottles secured incorrectly - fall off before or during experiment.	Potentially hazardous ice-bath contents. Runined sample(s).	Human error.	6	MI and SOP	9	8	432	MI to specify final check to ensure sample bottles are secured correctly.	6	8	3	144
	No quenching heel in sample bottle.	High pH of final sample, causing invalid results.	Human error.	3	MI and SOP	3	3	27	None	3	3	3	27

PROCESS: HYDROLYSIS OF ENERGETIC MATERIALS (Manufacturing Instructions)					ASSESSORS: A.Wilson, G.Plum, A.Lemke.					ESTIMATED IMPACT			
Ref.	POTENTIAL FAILURE MODE	POTENTIAL EFFECTS	POTENTIAL CAUSES	[S]	CURRENT CONTROLS	[O]	[D]	RPN	RECOMMENDED ACTION	[S]	[O]	[D]	RPN
	Too much quenching heel in sample bottle.	Invalid testing results.	Human error.	3	MI and SOP	3	3	27	None	3	3	3	27
	Incorrect quenching chemical added to sample bottle.	Incompatibility, possible gassing / reaction leading to personnel exposure.	Human error.	8	Trained Laboratory technicians, familiar with sample preparation.	2	8	128	None	8	2	8	128
2.2.2.8	Water batch not cranked up high enough.	Samples not chilled adequately.	Human error.	5	MI and SOP	5	6	150	None	5	5	6	150
2.2.3	Valves 201-A and 201-B closed or partially closed.	Vapors enter scrubber. Concentrations greater than expected.	Human error.	3	Scrubber capable of coping with significant effluent. MI and SOP.	5	8	120	None	3	5	8	120
2.2.4	Valve A-1 closed or partially open	Reduced or no acid flow during neutralization process step.	Human error.	1	Neutralization stage delayed until valve opened. No significant issue.	5	3	15	None	1	5	3	15
2.2.5	Valve C-1 closed or partially open	Reduced or no caustic flow during process. Reaction will not proceed.	Human error.	5	Caustic flow, pH monitored by PLC. Explosive feed will not be permitted by PLC. No significant	3	6	90	None	5	3	6	90

PROCESS: HYDROLYSIS OF ENERGETIC MATERIALS (Manufacturing Instructions)					ASSESSORS: A.Wilson, G.Plum, A.Lemke.					ESTIMATED IMPACT			
Ref.	POTENTIAL FAILURE MODE	POTENTIAL EFFECTS	POTENTIAL CAUSES	[S]	CURRENT CONTROLS	[O]	[D]	RPN	RECOMMENDED ACTION	[S]	[O]	[D]	RPN
					issue.								
2.2.6	Air sample valve 100 not open or partially open	Poor gas samples, ruining experiment	Human error.	3	MI and SOP	5	6	90	None	3	5	6	90
2.2.7	Valve 23-1 open or partially open	Dump tank contents can be pumped to reactor (requires a second failure).	Human error.	3	Reactor contents would overflow back into dump tank.	2	6	36	None	3	2	6	36
2.2.8	Valve AS-1 Closed or partially open.	Reactor contents seep into agitator seal, leaving an energetic residue which ignites at a later time.	Human error.	8	MI and SOP	5	6	240	MI to specify check for water discharge on north side of reactor, confirming flow.	8	5	3	120
2.2.9	Valve R-1 Closed or partially open.	No / reduced recirculation of reactor contents during	Human error.	2	PLC detects flow in recirculation line and initiates auto-shut down.	3	3	18	None	2	3	3	18

PROCESS: HYDROLYSIS OF ENERGETIC MATERIALS (Manufacturing Instructions)					ASSESSORS: A.Wilson, G.Plum, A.Lemke.					ESTIMATED IMPACT			
Ref.	POTENTIAL FAILURE MODE	POTENTIAL EFFECTS	POTENTIAL CAUSES	[S]	CURRENT CONTROLS	[O]	[D]	RPN	RECOMMENDED ACTION	[S]	[O]	[D]	RPN
		operation.											
2.2.10	Valve 201-C closed or partially open.	No reactor cooling, leading to heat build up in reactor and reaction runaway.	Human error.	2	PLC detects and informs operator of temperatures. MI and SOP. Auto dumping of reactor in event of over temperature.	5	6	60	None	2	5	6	60
2.2.11	Valve ST-1 on Sample Line closed or partially open.	Tracer line not heated, resulted in incorrect samples taken during experiment.	Human error.	3	MI and SOP	5	8	120	None	3	5	8	120
2.2.12	Valve ST-1 supplying steam to heat exchanger closed or partially open.	Heat exchanger unable to heat reactor contents during operation. Experiment delayed.	Human error.	1	PLC checks reactor temperature during operation.	8	3	24	None	1	8	3	24

PROCESS: HYDROLYSIS OF ENERGETIC MATERIALS (Manufacturing Instructions)					ASSESSORS: A.Wilson, G.Plum, A.Lemke.					ESTIMATED IMPACT			
Ref.	POTENTIAL FAILURE MODE	POTENTIAL EFFECTS	POTENTIAL CAUSES	[S]	CURRENT CONTROLS	[O]	[D]	RPN	RECOMMENDED ACTION	[S]	[O]	[D]	RPN
2.2.13	Valve 201-D closed or partially open.	No reactor cooling, leading to heat build up in reactor and reaction runaway.	Human error.	2	PLC detects and informs operator of temperatures. MI and SOP. Auto dumping of reactor in event of over temperature.	5	6	60	None	2	5	6	60
2.2.14	Valve HV-7-1 open or partially open	Reactor contents leak during experiment, resulting in dump tank being partially filled with hydrolysate / caustic, and failed experiment. Potential for personnel exposure.	Human error.	8	MI and SOP	5	8	320	Fit sensor to valve and link to PLC; program such that reactor cannot operate whilst valve is not fully closed.	8	5	1	40
2.2.15	Valve 19-1 open or partially open.	Would change reactor dumping cycle and may redirect material to tank farm.	Human error.	2	PLC checks and acts upon status of valve prior to operation of reactor.	3	3	18	None	2	3	3	18

PROCESS: HYDROLYSIS OF ENERGETIC MATERIALS (Manufacturing Instructions)					ASSESSORS: A.Wilson, G.Plum, A.Lemke.					ESTIMATED IMPACT			
Ref.	POTENTIAL FAILURE MODE	POTENTIAL EFFECTS	POTENTIAL CAUSES	[S]	CURRENT CONTROLS	[O]	[D]	RPN	RECOMMENDED ACTION	[S]	[O]	[D]	RPN
		Partial reactor contents would fill hydrolysate line to tank farm during dump cycle. Potential for partial, reduced-rate hydrolysis reaction in hydrolysate line (unheated, uninsulated line). Not considered a significant hazard.	Mechanical failure of valve (leaking)	6	Preventative Maintenance	5	8	240	None	6	5	8	240
	Valve 42 open or partially open.	Would change reactor dumping cycle and may redirect material to loading dock.	Human error.	2	PLC checks and acts upon status of valve prior to operation of reactor.	3	3	18	None	2	3	3	18



PROCESS: HYDROLYSIS OF ENERGETIC MATERIALS (Manufacturing Instructions)					ASSESSORS: A.Wilson, G.Plum, A.Lemke.					ESTIMATED IMPACT			
Ref.	POTENTIAL FAILURE MODE	POTENTIAL EFFECTS	POTENTIAL CAUSES	[S]	CURRENT CONTROLS	[O]	[D]	RPN	RECOMMENDED ACTION	[S]	[O]	[D]	RPN
		Partial reactor contents would fill hydrolysate line to loading dock during dump cycle. Potential for partial, reduced-rate hydrolysis reaction in hydrolysate line (boxway heated, uninsulated line). Not considered a significant hazard.	Mechanical failure of valve (leaking)	6	Preventative Maintenance	5	8	240	None	6	5	8	240
	Valve HV-12-2 open or partially open	No impact on hydrolysis process, would delay hydrolysis line cleaning only.	Human error.	2	MI and SOP	5	6	60	None	2	5	6	60

PROCESS: HYDROLYSIS OF ENERGETIC MATERIALS (Manufacturing Instructions)					ASSESSORS: A.Wilson, G.Plum, A.Lemke.					ESTIMATED IMPACT			
Ref.	POTENTIAL FAILURE MODE	POTENTIAL EFFECTS	POTENTIAL CAUSES	[S]	CURRENT CONTROLS	[O]	[D]	RPN	RECOMMENDED ACTION	[S]	[O]	[D]	RPN
	Valve 12-1 closed or partially open.	Would prevent reactor dumping via bottom of reactor valve.	Human error.	2	PLC checks and acts upon status of valve prior to operation of reactor. Reactor has two other independent means of emptying into dump tank.	3	3	18	None	2	3	3	18
2.2.16	Valve ST-3 closed or partially open.	No / reduced steam heating of gas sample line, leading to incorrect gas sample.	Human error.	3	MI and SOP	5	8	120	None	3	5	8	120
2.2.17	Dump tank filled with too much water	Dump tank contents would overflow to catch-basin.	Human error.	5	Dump tank level indicator linked to PLC. Reactor will not start unless tank only partially filled. MI and SOP	5	6	150	None	5	2	3	30
	Dump tank filled with too little water	Reactor contents would not be adequately quenched, possible	Human error.	9	Dump tank level indicator linked to PLC. Reactor will not start unless tank only partially filled. MI and SOP	5	6	270	None	9	2	3	54

PROCESS: HYDROLYSIS OF ENERGETIC MATERIALS (Manufacturing Instructions)					ASSESSORS: A.Wilson, G.Plum, A.Lemke.					ESTIMATED IMPACT			
Ref.	POTENTIAL FAILURE MODE	POTENTIAL EFFECTS	POTENTIAL CAUSES	[S]	CURRENT CONTROLS	[O]	[D]	RPN	RECOMMENDED ACTION	[S]	[O]	[D]	RPN
		runaway reaction in dump tank.											
	Dump tank filled with liquid other than water (e.g. hydrolysate)	No significant impact - hydrolysate solution essentially as capable of quenching reaction as water.	Human error.	2	MI and SOP	3	8	48	None	2	3	8	48
2.2.18	Valve AS-2 closed or partially open.	Dump tank contents seep into dump tank seal, leading to an ignition at a later time.	Human error.	1	PLC checks water flow - auto shut down if flow drops below a pre-defined minimum. MI and SOP.	3	6	18	None	1	3	6	18
2.2.19	Scrubber flow water valve SW-1 closed or partially open.	Reactor gas emissions not scrubbed. Potential for gas emissions	Human error.	5	PLC checks for water flow and signals if low. MI and SOP.	5	3	75	None	5	5	3	75

PROCESS: HYDROLYSIS OF ENERGETIC MATERIALS (Manufacturing Instructions)					ASSESSORS: A.Wilson, G.Plum, A.Lemke.					ESTIMATED IMPACT			
Ref.	POTENTIAL FAILURE MODE	POTENTIAL EFFECTS	POTENTIAL CAUSES	[S]	CURRENT CONTROLS	[O]	[D]	RPN	RECOMMENDED ACTION	[S]	[O]	[D]	RPN
2.2.20	Valve 41 closed or partially open.	Reduced / no overflow capability from reactor to dump tank. Potential for reactor flooding thro' scrubber during dump cycle (requires at least one other failure).	Human error.	5	PLC checks that valve is open. Reactor cannot start unless valve position is correct. MI and SOP	5	3	75	None	5	5	3	75
2.2.21	Valve 10-3 off or partially on.	Reduced reactor cooling capability, leading to over temperature during process.	Human error.	3	Reactor dump cycle and thermal inertia of reactor. Not expected to result in any major safety issues.	5	8	120	None	3	5	8	120
2.2.22	Valve HS 10-2 in incorrect position.	Potential for heating of dump tank, reducing the effectiveness of	Human error.	1	PLC detects dump tank temperature. MI and SOP	5	6	30	PLC to flag an alarm if dump tank temperature exceeds 40°C	1	5	3	15

PROCESS: HYDROLYSIS OF ENERGETIC MATERIALS (Manufacturing Instructions)					ASSESSORS: A.Wilson, G.Plum, A.Lemke.					ESTIMATED IMPACT			
Ref.	POTENTIAL FAILURE MODE	POTENTIAL EFFECTS	POTENTIAL CAUSES	[S]	CURRENT CONTROLS	[O]	[D]	RPN	RECOMMENDED ACTION	[S]	[O]	[D]	RPN
		the dump mode, but not the dump capability.											
2.2.23	Valve D-2 open or partially open.	Dyked area would not automatically drain to dump tank in the event of a spillage.	Human error.	1	MI and SOP	3	2	6	None	1	3	2	6
2.2.24	Valve D-1 open or partially open	Dump tank contents would leak into hydrolysate lines. Potential for hydrolysates to mix.	Human error.	5	MI and SOP	8	6	240	None	5	8	6	240
2.2.25	Valve C-2 closed or partially closed.	Unable to load caustic to tank farm	Human error.	1	MI and SOP	5	3	15	None	1	5	3	15
	Valve C-3 closed or partially closed.	Unable to load caustic into reactor.	Human error.	1	MI and SOP	5	3	15	None	1	5	3	15
	Valve C-4 closed or partially closed.	Unable to load caustic into reactor.	Human error.	1	MI and SOP	5	3	15	None	1	5	3	15



**TABLE 3 – DEFINITIONS FOR THE FMEA SEVERITY, OCCURRENCE AND DETECTABILITY VALUES**

No.	DEFINITION		
	SEVERITY [S]	OCCURRENCE [O]	DETECTABILITY [D]
1	No significant impact (safety, cost or timescale).	Never been known to occur anywhere.	Obvious; cannot easily be missed by staff likely to be present.
2	Some administration issues, minor cost impact.	Never been known to occur at HSAAP based on past performance; considered unlikely to occur in process.	Not used
3	Minor schedule impact	Similar occurrence known to have occurred at least once at HSAAP in the past, but considered rare.	Detectable by a casual inspection only
4	Not used	Not used	Not used
5	Program delays expected. Moderate cost impact likely.	Occurred more than once in the past at HSAAP in a similar activity – a realistic but probably unlikely occurrence for the process.	Not used
6	Expect equipment damage. Potential for minor personnel exposure	Not used	Detectable as part of the standard process inspection / monitoring only.
7	Major cost and/or schedule delay; no significant personnel safety issues	Not used	Not used
8	Expect minor personnel health risks and/or significant equipment damage / cost / delay	Expected to occur at least once during the planned experimental trials for the program.	Detectable only if specifically looked for; will probably not otherwise be detected.
9	Imminent danger to personnel and/or substantial equipment / facility damage and/or cost/delays. Very unsafe.	Expected to occur more than once during the planned experimental trials for the program.	Unlikely to find without an intensive specific search.
10	Catastrophic event to personnel and/or damage to facility. Prohibitive cost / schedule impact likely	Frequent occurrence during the life of the program expected.	Probably undetectable until too late; cannot reasonably be expected to be found

**FMEA – MANUFACTURING INSTRUCTIONS**

1. The following text relates to the proposed procedure for operating the HYDROLYSIS FACILITY. The instructions have been evaluated in the Failure Modes and Effects Analysis (FMEA) reported herein. The instructions have been incorporated into the HYDROLYSIS FACILITY Manufacturing Instructions as part of the overall facility documentation.

**2. PRE-OPERATIONAL CHECKLISTS****2.1 Pre-operational Checklist Prior to Operating the Reactor - Building G-10 Tank Farm**

- 2.1.1 Manually close the valves (identified as HV-10, HV-12, HV-14, and HV-15) on the discharge lines of Tank Nos. GT-10, GT-12, GT-14, and GT-15 that are designated for storage of the hydrolysate product.
- 2.1.2 Manually open the valve (identified as No. HV-21-1) located on the discharge line of the GT-11 Caustic Storage Tank.
- 2.1.3 Manually close (or confirm closed) the valve (identified as HV-22-1) located on the discharge line of the GT-13 Acid Storage Tank.
- 2.1.4 Manually close the two dike drain valves located on the north side of the two diked areas in the Building G-10 Tank Farm using a tee-handle.
- 2.1.5 If upon visual inspection, liquid is visible inside the diked area then inform supervision who will determine whether the liquid is the result of a leaking tank or simply water from rainfall or steam condensate. If any observed liquid is determined to be simply rainfall or steam condensate then open the two-dike drain valves to drain water to the industrial sewer. Close the valves immediately upon completion of this operation.

**2.2 Pre-operational Checklist Prior to Operating the Reactor - Inside Building G-10**

- 2.2.1 Manually open (or confirm open) the valve (identified as No. FV-1 and located on the third floor {west side} of Building G-10 adjacent to the material lift bay) which supplies water to the system deluge nozzle above the feed hopper.
- 2.2.2 Isolate the sampler to prevent remote operation by switching the SAMPLER E-STOP lever to the off position. The E-STOP lever is located on the west wall adjacent to the sampler in Building G-10. The remote sampling cabinet can now be prepared as follows:
  - 2.2.2.1 Close the drain valve located on the ice water bin in the sample cabinet.
  - 2.2.2.2 Remove the screen from the sample tray in the sample cabinet.
  - 2.2.2.3 Load the sample bin to the top of the screen support with ice, and replace the screen.
  - 2.2.2.4 Install the four latches to secure the screen.
  - 2.2.2.5 Place ice in the center compartment on top of the screen.



- 2.2.2.6** Using the water hose mounted on Column C-3 in the west side of Building G-10, manually add water to the sample bin filled with ice until the level reaches indicator mark approximately one inch from the top lip of the ice bin.
- 2.2.2.7** Load the new sample bottles, containing the pre-measured acid heel for quenching, into the sample carousel. (Make sure all bottles are threaded snugly into the teflon bottle holders.)
- 2.2.2.8** Using the manual crank, lift the water bath to its maximum height which is at an appropriate level to partially cover the sample bottles in the ice water.
- 2.2.2.9** Close the door(s) of the sample cabinet and secure with the latches.
- 2.2.2.10** Switch the SAMPLER E-STOP lever to the ON position to activate the sampler thereby enabling computer control.
- 2.2.3** Manually open the two valves (identified as Nos. 201-A and 201-B) on the line providing cooling water to the condenser located on the north side of the reactor skid (upper level) opposite of the manhole cover.
- 2.2.4** Manually open the valve (identified as No. A-1) installed in series with the automated valve on the acid feed line located on the east side of the reactor skid (upper level).
- 2.2.5** Manually open the valve (identified as No. C-1) installed in series with the automated valve on the caustic feed line located on the east side of the reactor skid (upper level).
- 2.2.6** Manually open the air sample valve (identified as No. 100) on the port on the gas sampling valve located on the east side of the reactor skid (upper level).
- 2.2.7** Manually close the valve (identified as No. 23-1) on the pipe allowing dump tank contents to be pumped back to the reactor (located on the east side of the reactor skid (upper level) adjacent to the manhole cover).
- 2.2.8** Manually open the valve (identified as AS-1) providing water to the water seal of the agitator located on the south side of the reactor skid (upper level) next to the manhole cover. The flow of water should be adjusted to achieve maximum flow without overflowing the seal. Open the manhole cover and visually confirm that water is not leaking into the reactor. Also, check the discharge pipe (labeled as Agitator Seal Water) at the bottom of the reactor skid (north side) to ensure water is flowing.
- 2.2.9** Manually open the valve (identified as No. R-1) on the circulation loop of the reactor that is located adjacent to the manhole on the reactor.
- 2.2.10** Manually open the valve (identified as No. 201-C) on the line providing cooling water to the heat exchanger located on the north side of the reactor skid (lower level) opposite of the manhole cover.
- 2.2.11** Manually open (or confirm opened) the valve (identified as No. ST-1) supplying steam to the tracer line on Sample Valve No. 100 located on the north side of the reactor skid (lower level).
- 2.2.12** Manually open the valve (identified as ST-1) on the line supply steam to the reactor heat exchanger located on the north side of the reactor skid (lower level).

- 2.2.13** Manually open the valve (identified as No. 201-D) on the line allowing flow of water from the jacket on the reactor to the industrial sewer located on the west side of the reactor skid (lower level).
- 2.2.14** Manually close (or confirm closed) the valve (identified as No. HV-7-1) on the recirculation pump suction line, located on the south side of the reactor skid at ground level. (This valve will later be used to collect the final sample of hydrolysate product at the end of a reactor run.)
- 2.2.15** On the discharge side of the hydrolysate transfer pump (No. PU4), manually close Valve Nos. 19-1, HV-42, and HV-12-2. Also, at this position, manually open Valve No. HV-12-1 on the overflow line between the reactor and the dump tank.
- 2.2.16** Manually open the valve (identified as No. ST-3), located adjacent to Valve No. 19-1 at floor level, that supplies steam to the steam tracer line on the gas sampling line coming from the reactor.
- 2.2.17** Inspect the water level in the dump tank. If necessary, adjust the level to the top of the interior heating / cooling coils (approximately 2000 gallons).
- 2.2.17.1** If water needs to be added, open the valve (identified as No. W-1) on filter water inlet line to the dump tank (located on the west side of the dump tank adjacent to the manhole).
- 2.2.18** Manually open the valve (identified as No. AS-2) providing water to the water seal on the dump tank (located on the west side of the top of the dump tank). Visually confirm water flow by inspecting the drain line outlet in the drain at the north wall at the back of the dump tank.
- 2.2.19** Manually open the scrubber water valve (identified as No. SW-1) and set the water flow at 40 gallon per minute (gpm)  $\pm$  5 gpm.
- 2.2.20** Manually open the valve (identified as No. HV-41) located on the overflow line (first floor) between the reactor and the dump tank.
- 2.2.21** Turn on the river water flow valve (identified as 10-3) using hand-spring switch No. HS 10-3 (located on the first floor on the north side of the dump tank). The hand –spring switch No. HS 10-3 should be in the “Off” (“down”) position.
- 2.2.22** Turn off hand-switch valve No. HS 10-2 (i.e. switch in down position) located on the first floor on the south side of the dump tank. Valve No. 10-2 switches between steam-on the water and out of the dump tank heating / cooling coils.
- 2.2.23** Manually close (or confirm closed) the valve (identified as No. D-2), which allows liquid within the dyked area around the reactor to be drained to the dump tank, located on the south side of the dump tank (first floor).
- 2.2.24** Manually close (or confirmed closed) the valve (identified as No. D-1) located on the bottom of the dump tank that controls the flow of dump tank contents.
- 2.2.25** Manually close Valve No. C-2 and manually open Valve Nos. C-3 and C-4, which control the direction of flow in the caustic transfer lines, located on the first floor (west side) of Building G-10.

### **2.3 Pre-operational Checklist Prior to Operating the Reactor - In Building E-10 Control Room**

- 2.3.1** Note: the pre-operational activities in E-10 can be undertaken whilst Building G-10 is manned.
- 2.3.2** Switch on the computer and log in.
- 2.3.3** Adjust the remote feeder setpoints to feed the reactor with the desired quantity of energetic material (maximum 500 LB) at the desired feed rate as specified on the batch sheet.
- 2.3.4** Enter the desired reaction digest time into the PLC via the computer.
- 2.3.5** Click on the “START BATCH” icon in the computer screen to initiate a batch startup.
- 2.3.6** Check to ensure that the reactor tank level is less than 10% (which could mean zero) and that the measured temperature inside the reactor is less than 90°C. If either of these conditions are not true then consult with supervision.
- 2.3.7** Add water to the reactor (quantity as specified on the batch sheet).
- 2.3.8** Apply heating to the reactor, set point  $85\pm 2^{\circ}\text{C}$  unless otherwise specified on the batch sheet.

At this point, the reactor is ready for operation. Energetic materials are loaded into the loss in weight feeder, the building is checked and the reaction is conducted using the PLC. Full details are given in the Manufacturing Instruction.

## **APPENDIX K**

### **Off Gassing Analytical Procedures**

## PM ACWA ENERGETIC HYDROLYSATE PRODUCTION HOLSTON AAP SITE GAS SAMPLING OVERVIEW

TRC's role on the PMACWA Energetic Hydrolysate program being conducted at the Holston Army Ammunition Plant is to quantify the gaseous components being released from the hydrolyzation process. In order to accomplish this goal, an integrated gas sample is withdrawn from the 01 position (or the exhaust vent) of the Reactor Vessel and transported to the on-site analytical laboratory operated by TRC. Two types of samples are then collected in that laboratory: 1) Near real-time sampling of gaseous components that are conducive to analysis by Continuous Emission Monitoring System (CEMS); and, 2) Batch samples that are collected using an adsorption media or absorbing solution that are recovered and sent to an off-site laboratory for final quantification. The CEMS analyzers provide minute-minute trends of the concentrations of the individual gaseous compounds while the batch trains yield one value that represents an average concentration for the entire test run. Table 1 details the individual test methods that TRC is using in support of the PMACWA test program.

**Table 1. , Test Methods**

<i>Parameter</i>	<i>Method</i>	<i>Abbreviation</i>	<i>Media Type</i>	<i>Laboratory</i>
Oxygen/Carbon Dioxide	EPA Method 3A	O <sub>2</sub> /CO <sub>2</sub>	CEMS	On-site
Moisture	EPA Method 4	Bws	Gravimetric	On-site
Nitrogen Oxides	EPA Method 7E	NO <sub>x</sub>	CEMS	On-site
Carbon Monoxide	EPA Method 10	CO	CEMS	On-site
Total Hydrocarbons	EPA Method 25A	THC	CEMS	On-site
Aldehyde/ketones	SW-846 Method 0011	Ald/Ket	DNPH	DAT, Inc.
Ammonia/Hydrogen Cyanide	EPA CTM 027/M26A	NH <sub>3</sub> /HCN	H <sub>2</sub> SO <sub>4</sub> /NaOH	Philip Analytical
Energetic Materials	CHPPM STEM	STEM	Imp/XAD	CHPPM
Volatile Compounds	EPA TO-15	TO-15	SUMMA	Philip Analytical
Nitrous Oxide	NIOSH 6600	N <sub>2</sub> O	IR	On-site by Miran 1B2

The sampling apparatus is divided into the following functional groups or systems in order to collect these samples. The following items are described in the direction of the sample gas flow (from Reactor Vessel to the analytical collection system maintained in the trailer):

1. A 1-inch ID Teflon® sample probe is positioned in the gas stream at the exhaust vent of the Reactor Vessel immediately exit of the headspace of the vessel. This probe is connected to three separate Teflon® sample lines approximately 350 feet in length. The lines are steam traced inside the building (about 150 feet) and electrically traced once they exit the restricted area of the building (about 200 feet) and are maintained at approximately 225-250 °F in order to prevent condensation of moisture (or organic compounds) during transport. The three lines have the following function:

**Line 1 - The Batch Train sample line** – is a 0.5-inch ID Teflon sample line that is used to transport approximately 20 Liters/min of headspace gas to the individual batch trains.

**Line 2 – The CEMS Sample line** – is a 0.375-inch ID Teflon sample line that is used to transport approximately 10-15 Liters/min of headspace gas to the CEMS analyzers.

**Line 3 – The CEMS Calibration line** – is a 0.375-inch ID Teflon sample line that is used to transport calibration gas from the mobile laboratory to the sample valve and back to the CEMS analyzers in order to calibrate the analytical instruments.

2. The CEMS portion of the gas sample is directed to the back of the analytical trailer and is divided once it enters the mobile laboratory as follows:

- a. A small portion of the sample is maintained heated and is directed to the total hydrocarbon analyzer in order to analyze for total VOC content of the gas stream. It is important that this sample remains heated to prevent condensation of any organic species that may be present.
  - b. The bulk of the sample goes through a sample conditioning system that is designed to remove the moisture from the sample stream prior to analysis. The gas stream is then further split to the following analyzers for near real-time analysis and data collection: Oxygen (O<sub>2</sub>) Analyzer, Carbon Dioxide (CO<sub>2</sub>) Analyzer, Carbon Monoxide (CO) Analyzer, and the Nitrogen Oxides (NO<sub>x</sub> comprised of NO and NO<sub>2</sub>) Analyzer.
3. The output signal from each of the CEMS analyzers is recorded each second by the data acquisition system (DAS). From these data a 1-minute average is calculated and recorded by the DAS that can export the information to a Microsoft Excel file. Each analyzer is calibrated through the entire sample system before and after each test run using the calibration sample line. After the test run is completed the data are then corrected for calibration error or analyzer drift. The data are subjected to a series of quality assurance and quality control measures that are specified by the sampling methodology to verify the data is within precision and accuracy guidelines.
4. The Batch Train portion of the gas sample enters the front of the trailer and is directed to a multi-port Teflon® sample manifold that is maintained at approximately 250 °F. From that manifold the gas sample is split to the following sample trains:

**STEM Train** – The sampling train for energetic materials is a multi-impinger sample train maintained in chilled ice-bath followed by an adsorption media of XAD resin. The sample train is recovered on-site and sent to an off-site laboratory for analyses.

**Aldehyde/Ketone Train** – The sampling train for aldehyde/ketone compounds is a multi-impinger sample train filled with an absorbing solution containing a derivitizing agent maintained in a chilled ice-bath. The sample train is recovered on-site and sent to an off-site laboratory for analyses.

**Ammonia/Hydrogen Cyanide Train** – The sampling train for ammonia (NH<sub>3</sub>) and hydrogen cyanide (HCN) is a multi-impinger sample train filled with an absorbing solution maintained in a chilled ice-bath. The sample train is recovered on-site and sent to an off-site laboratory for analyses.

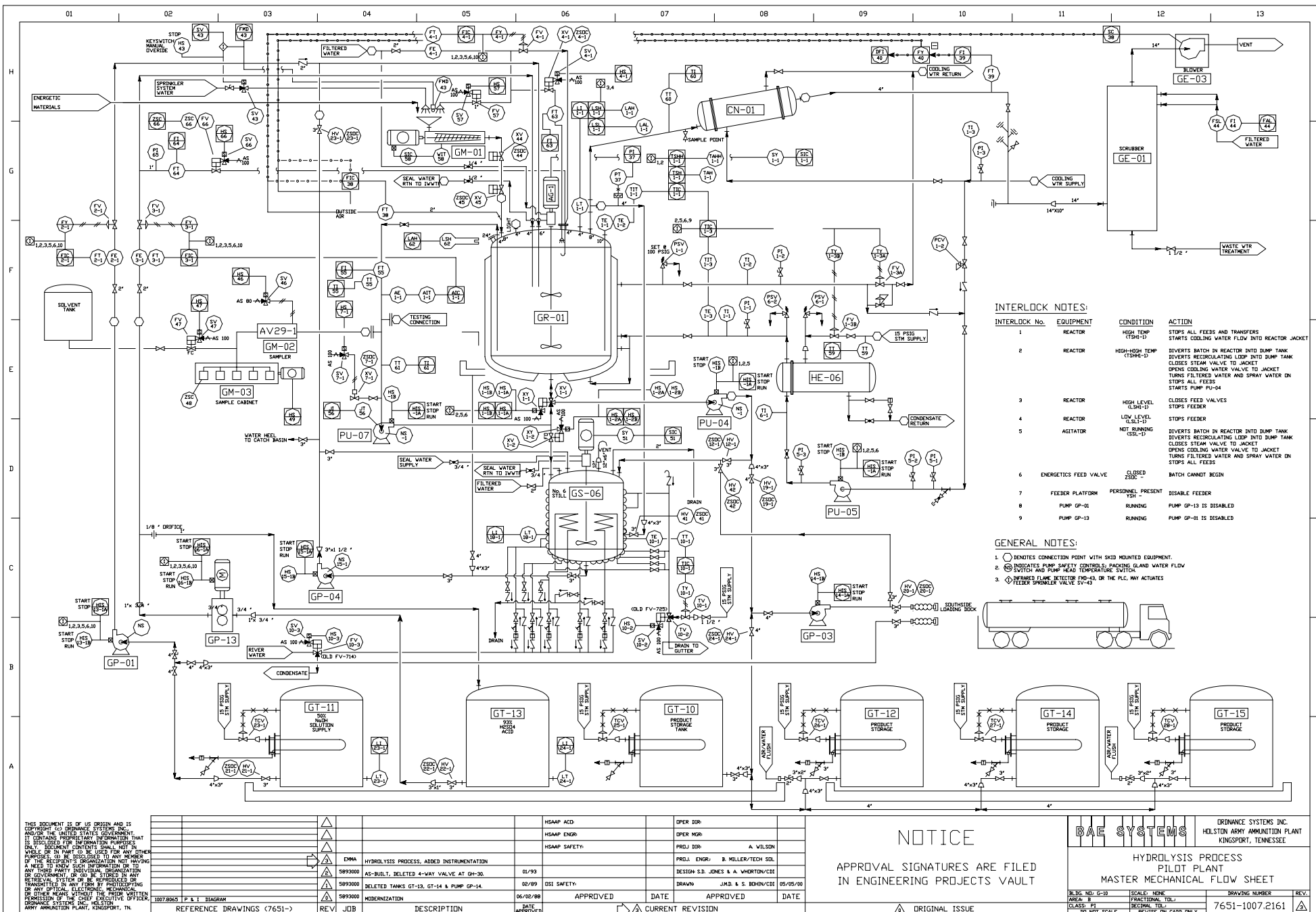
**VOCs** – An evacuated SUMMA canister is used to collect an integrated sample over the test period to be analyzed for a specific set of volatile organic compounds. The sample is sealed and sent to an off-site laboratory for analyses.

**Nitrous Oxide** – An integrated sample is withdrawn from the sample manifold and directed to a continuous analyzer to measure the N<sub>2</sub>O concentration. The N<sub>2</sub>O is measured real time and the data is logged into the DAS described in Item 3. This analyzer is operated from the Batch Train manifold rather than the CEMS because it requires a fairly large volume of sample gas.

5. The individual sample trains are transported to the Recovery Area at the conclusion of the sample run. Each train type goes through a specific methodology for recovering the absorbing solution or adsorbent media. After recovery of the train is completed the samples are sealed, uniquely labeled, and shipped under strict Chain-of-Custody to the laboratory for analyses. The collection data from the Batch Trains are recorded on field datasheets and entered into MS Excel spreadsheets to calculate the precise collection volume. This volume is used in conjunction with the specific compound mass determined by the laboratory to calculate a resultant gas stream concentration.

## **APPENDIX L**

### **System Installation Drawings**



NOTICE


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IN ENGINEERING PROJECTS VAULT

 ORIGINAL ISSUE

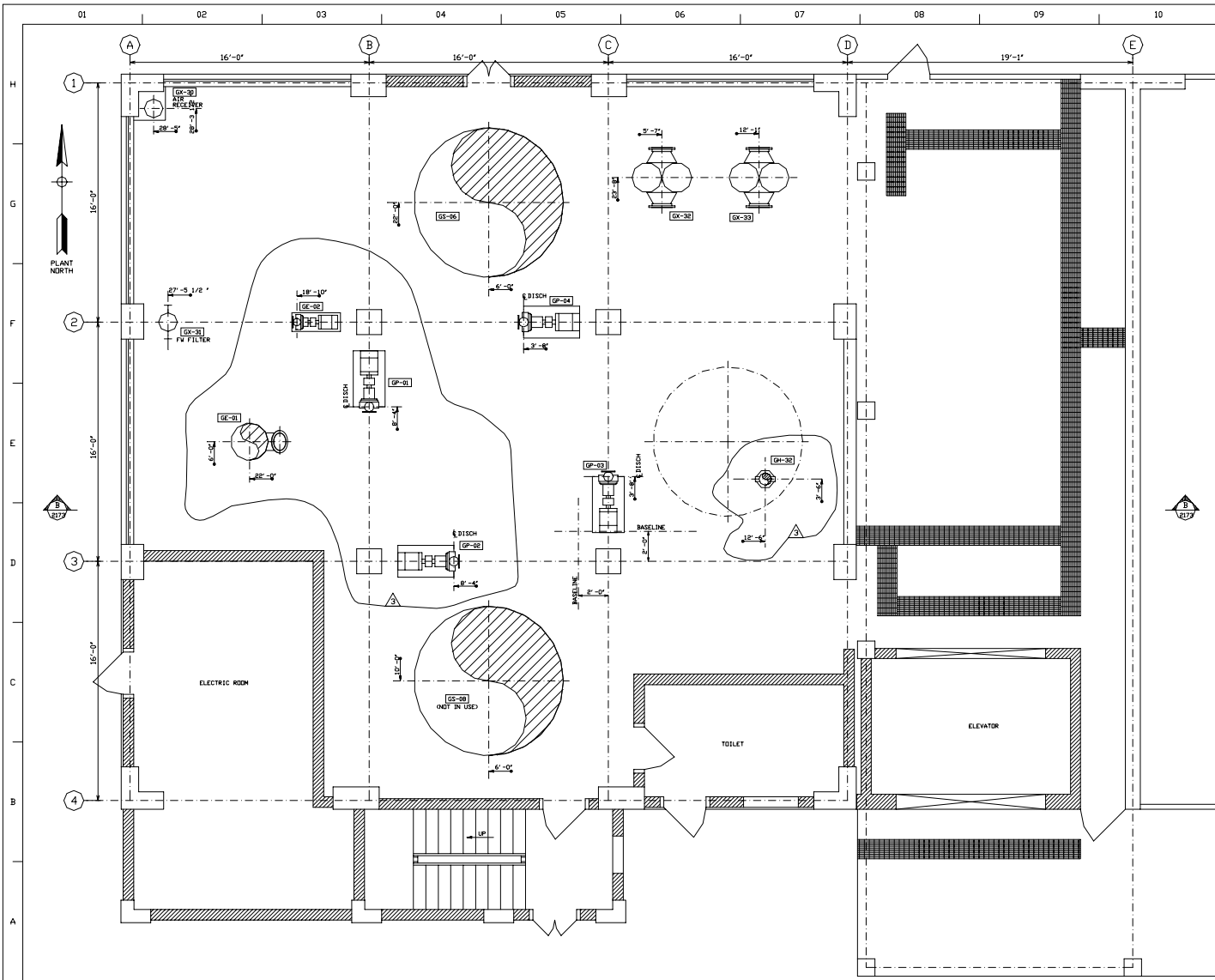
BAE SYSTEMS

ORDNANCE SYSTEMS INC.  
HOLSTON ARMY AMMUNITION PLANT  
KINGSPORT, TENNESSEE

HYDROLYSIS PROCESS  
PILOT PLANT  
MASTER MECHANICAL FLOW SHEET

BLDG. NO. G-10	SCALE: NONE	DRAWING NUMBER	REV.
AREA: B	FRACTIONAL TOL.	7651-1007.2161	
CLASS: P1	DECIMAL TOL.		
DO NOT SCALE	REVISE ON CARD ONLY		






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2	GS-08	STILL		EXISTING (OUT OF SERVICE)
3	GP-01	PUMP (NACH)		RE-INSTALL
4	GP-02	PUMP (CPAWD)		RE-INSTALL
5	GP-03	PUMP (HYDROLYSATE)		EXISTING
6	GP-04	PUMP (RECYCLE)		EXISTING
7	GE-01	SCRUBBER		RELOCATE FROM E-10 (GP-19)
8	GE-02	PUMP (SCRUBBER WATER)		RELOCATE FROM E-10 (ET-19)
9	GX-01	AIR RECEIVER		EXISTING
10	GX-02	FV FILTER		EXISTING
11	GX-03	RV STRAINER		EXISTING (OUT OF SERVICE)
12	GX-04	RV STRAINER		EXISTING
13	GH-02	STEAM CONDENSER		RELOCATE FROM E-10 (EX-00)

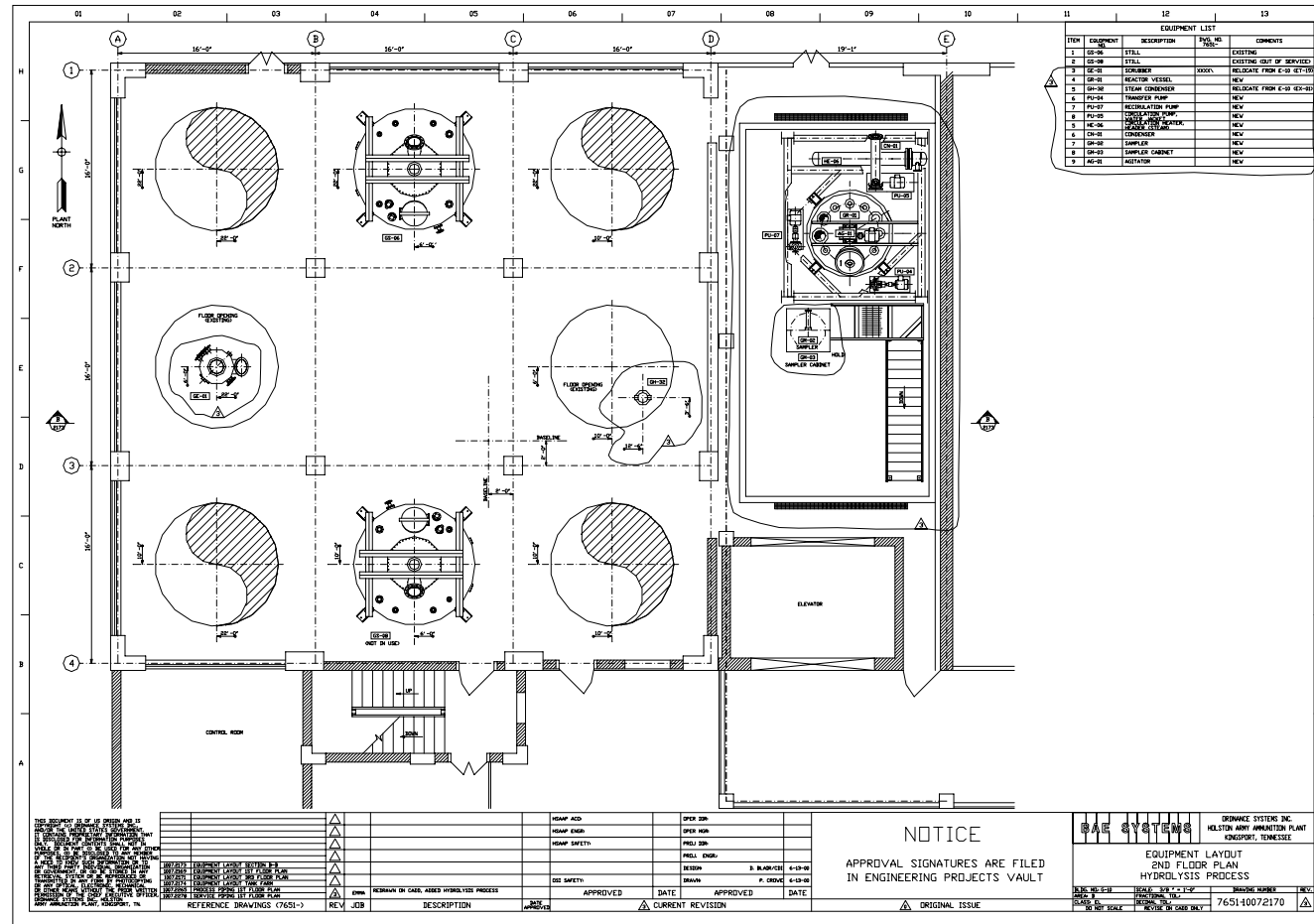
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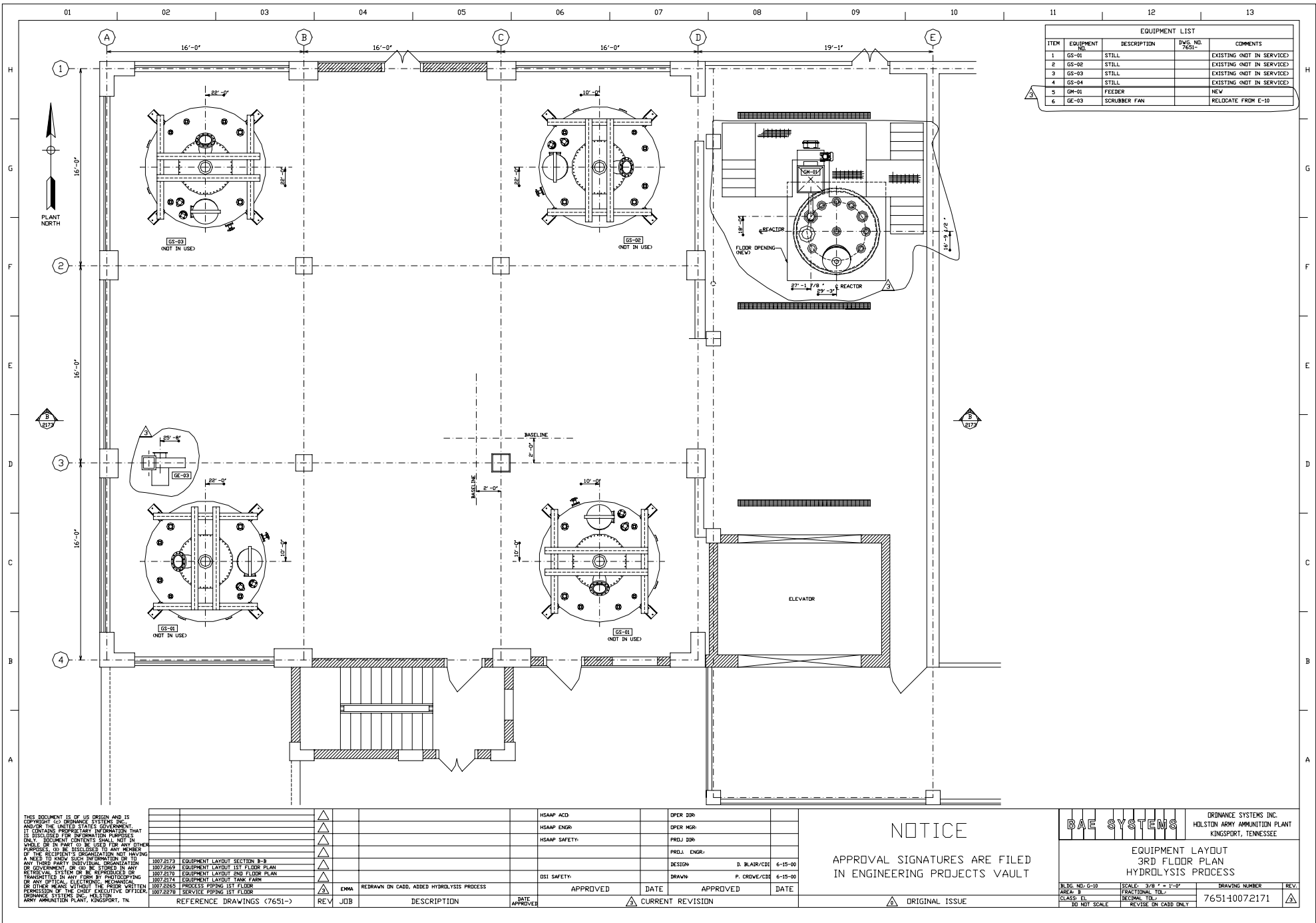
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10072175			EQUIPMENT LAYOUT 2ND FLOOR PLAN		
10072171			EQUIPMENT LAYOUT 3RD FLOOR PLAN		
10072174			EQUIPMENT LAYOUT TANK FARM PLAN		
10072265			PROCESS PIPING 1ST FLOOR PLAN		
10072278			SERVICE PIPING 1ST FLOOR PLAN		

HSAMP ACID	OPER. DIR.	
HSAMP ENGR.	OPER. MGR.	
HSAMP SAFETY	PROJ. DIR.	
	PROJ. ENGR.	
	DESIGN	D. BLAIR/CDE 6-15-00
	DRAWN	P. CRDVE/CDE 6-15-00
OSI SAFETY		
APPROVED	DATE	APPROVED
		DATE
		CURRENT REVISION

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ORIGINAL ISSUE

BAE SYSTEMS		ORDNANCE SYSTEMS INC. HOLSTON ARMY AMMUNITION PLANT KINGSFORT, TENNESSEE	
EQUIPMENT LAYOUT 1ST FLOOR PLAN HYDROLYSIS PROCESS			
BLDG. NO. G-10	SCALE: 3/8" = 1'-0"	DRAWING NUMBER	REV.
AREA 3	FRACTIONAL TOL.	7651-10072169	
CLASS. EL.	DECIMAL TOL.		
DO NOT SCALE	REVISE ON CASE ONLY		





EQUIPMENT LIST				
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2	GS-02	STILL		EXISTING (NOT IN SERVICE)
3	GS-03	STILL		EXISTING (NOT IN SERVICE)
4	GS-04	STILL		EXISTING (NOT IN SERVICE)
5	GS-05	FEEDER		NEW
6	GE-03	SCRUBBER FAN		RELOCATE FROM E-10


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REV	JOB	DESCRIPTION	DATE APPROVED	CURRENT REVISION
1		1007.2172 EQUIPMENT LAYOUT SECTION B-B		
2		1007.2169 EQUIPMENT LAYOUT 1ST FLOOR PLAN		
3		1007.2170 EQUIPMENT LAYOUT 2ND FLOOR PLAN		
4		1007.2174 EQUIPMENT LAYOUT TANK FARM		
5		1007.2265 PROCESS PIPING 1ST FLOOR		
6		1007.2278 SERVICE PIPING 1ST FLOOR		

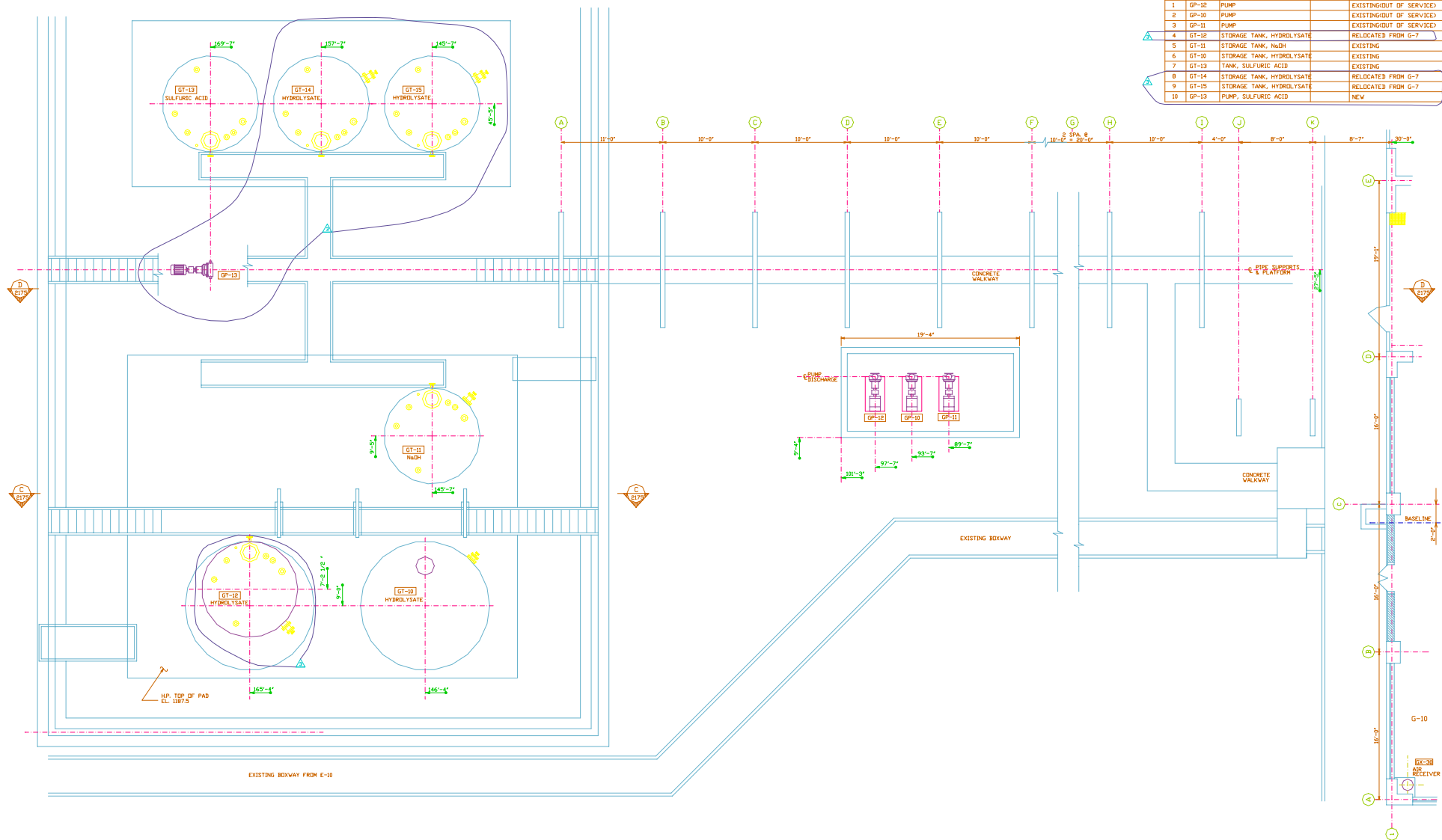
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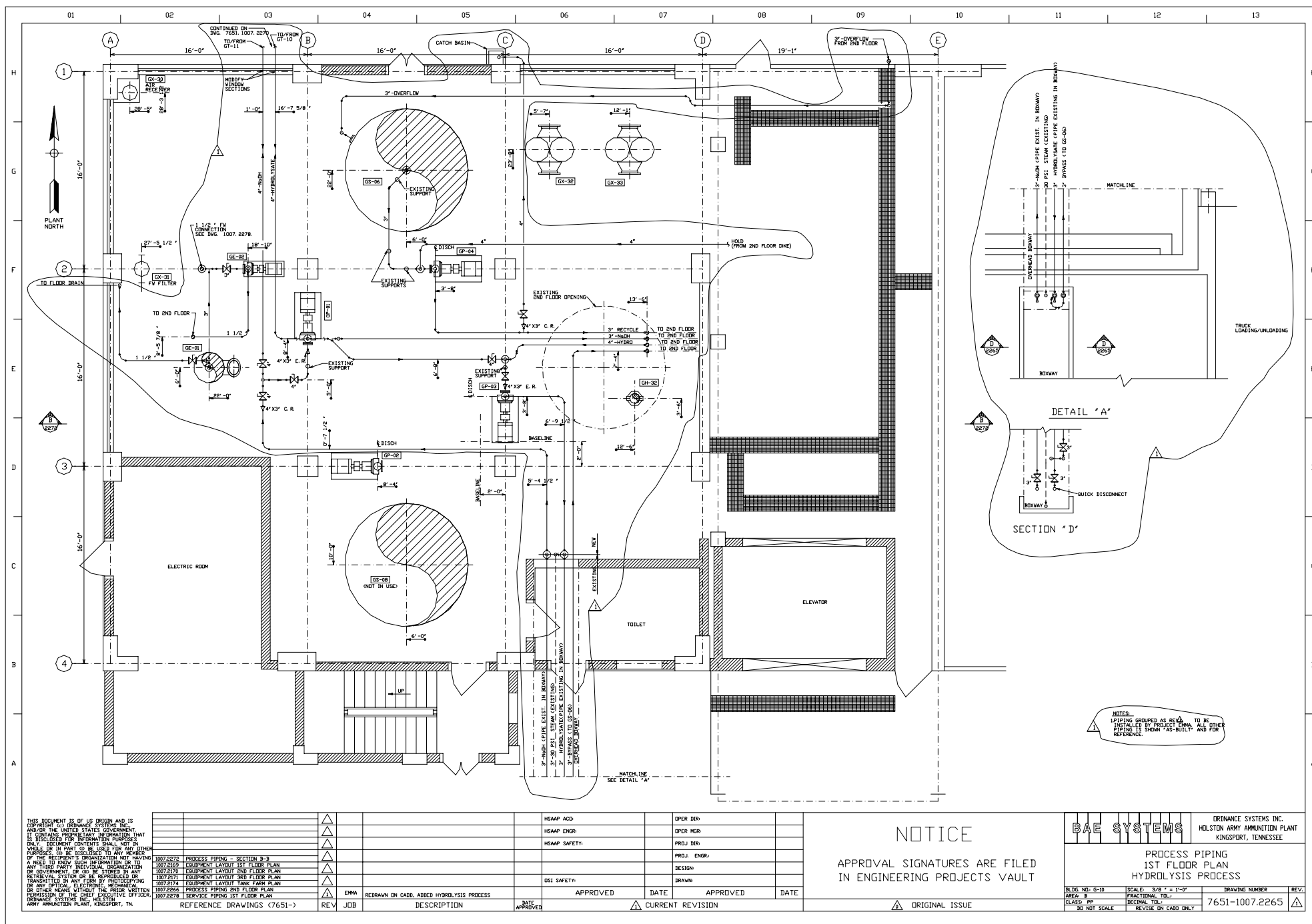
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HSAMP ENGR	OPER MGR
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	DRAWN P. CROVE/CDI 6-15-00

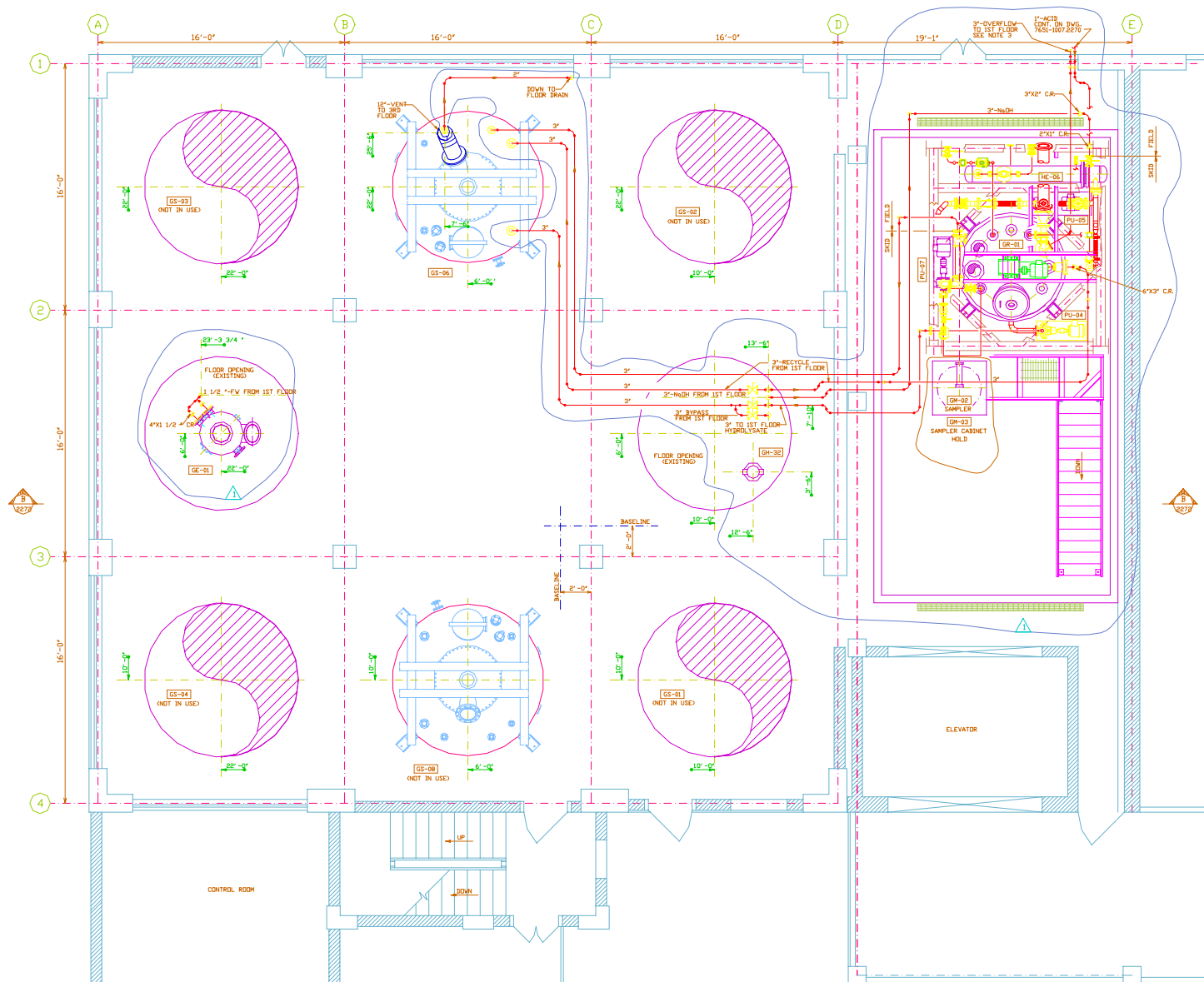
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BAE SYSTEMS		ORDNANCE SYSTEMS INC. HOLISTON ARMY AMMUNITION PLANT KINGSPORT, TENNESSEE	
EQUIPMENT LAYOUT 3RD FLOOR PLAN HYDROLYSIS PROCESS			
BUILDG. NO. G-10	SCALE: 3/8" = 1'-0"	DRAWING NUMBER	REV.
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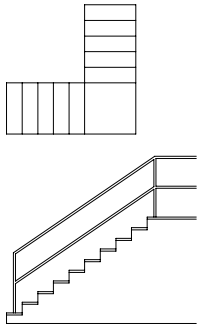
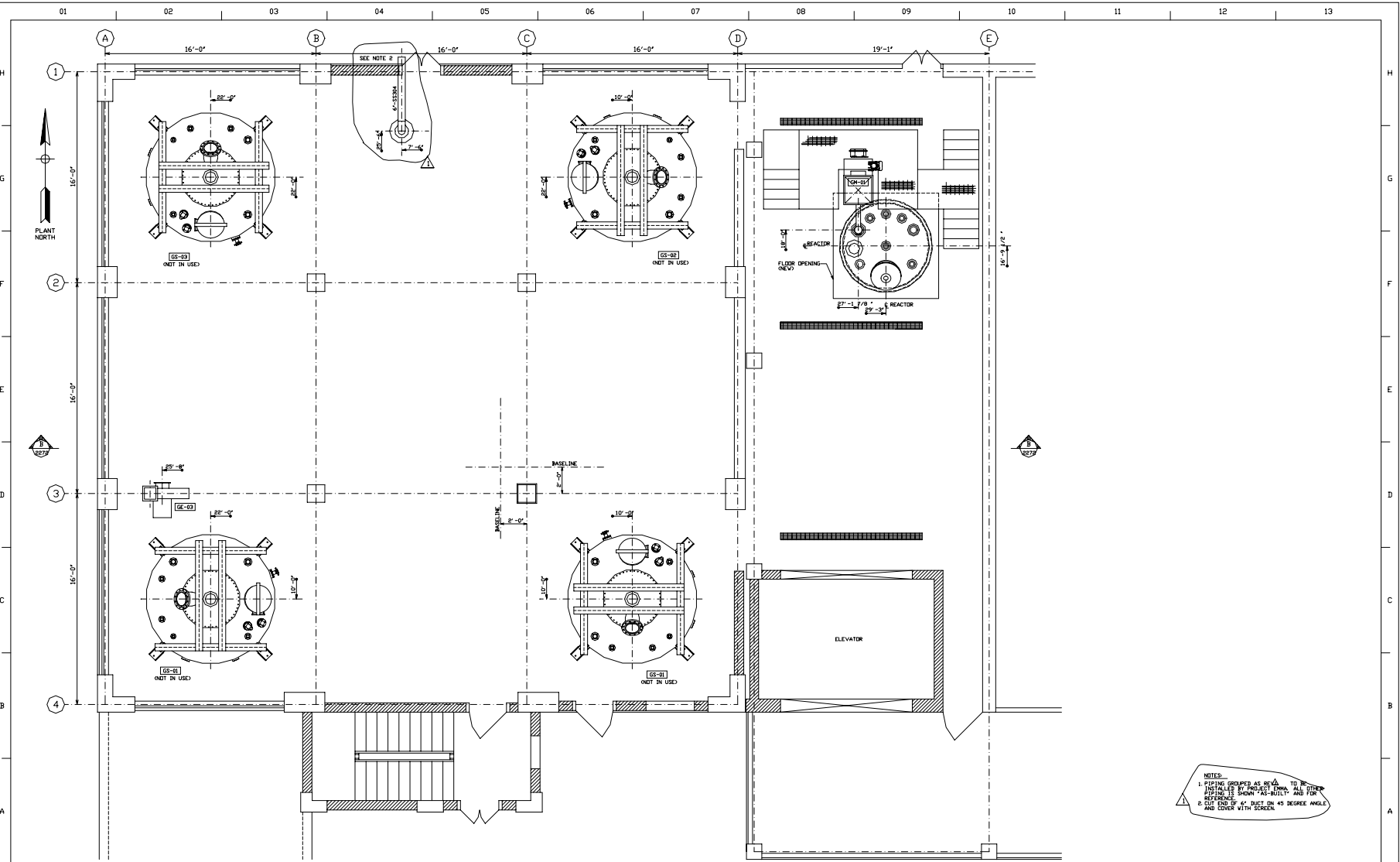








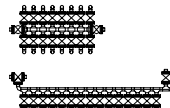
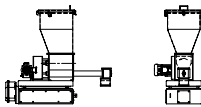
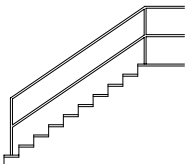
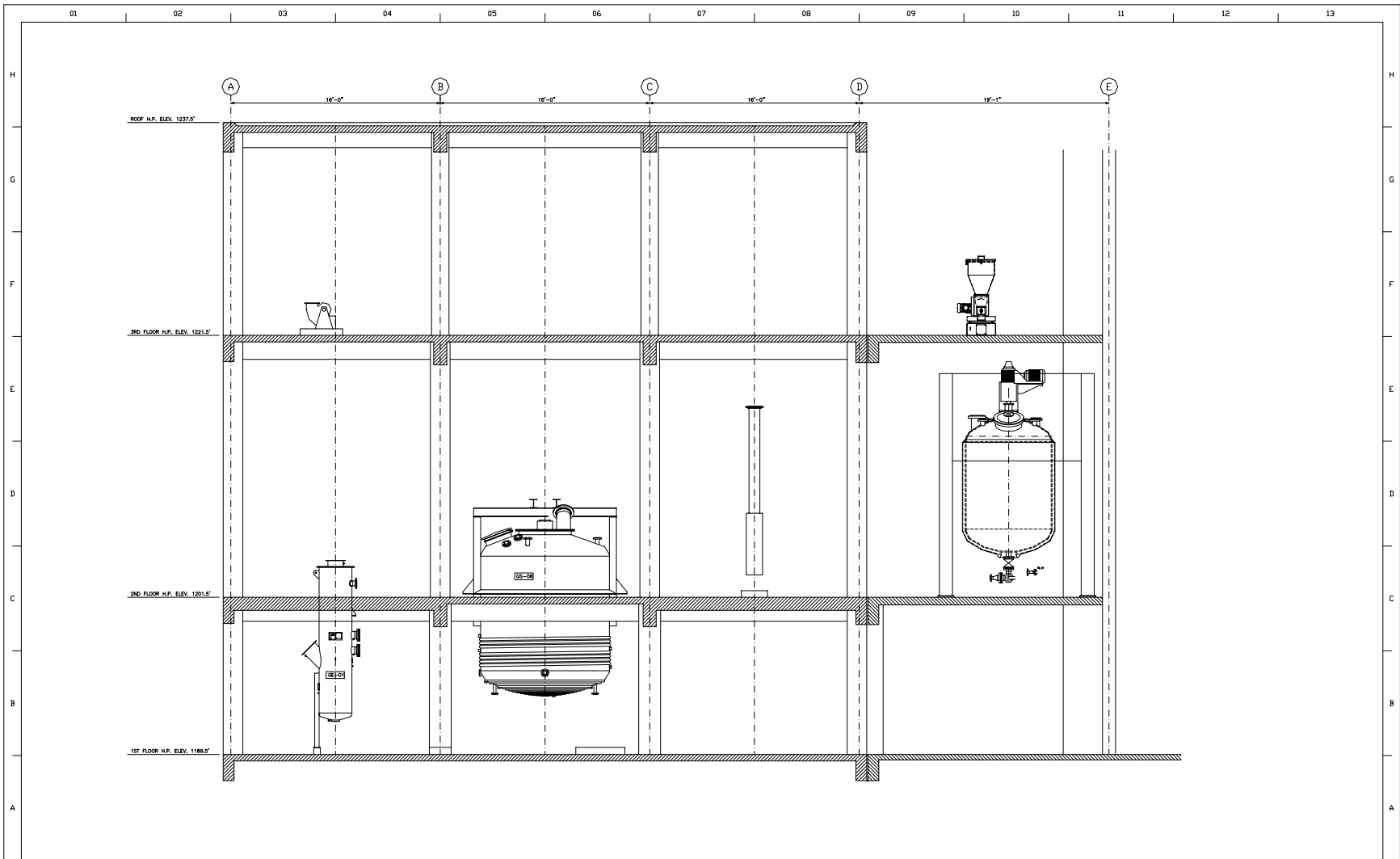
- NOTES:
1. PIPING GROUPED AS REV. A TO BE INSTALLED BY PROJECT CMA. ALL OTHER PIPING IS SHOWN "AS-BUILT" AND FOR REFERENCE.
  2. PIPING WITHIN REACTOR SKID ASSEMBLY TO BE SUPPLIED BY OTHERS. PROCESS PIPING TO BE INSTALLED TO DESIGNATED CONNECTION ON SKID ASSEMBLY.
  3. MINIMIZE OVERFLOW PIPE OUTSIDE BUILDING BETWEEN 1ST & 2ND FLOORS. INSULATE OUTSIDE PORTION.



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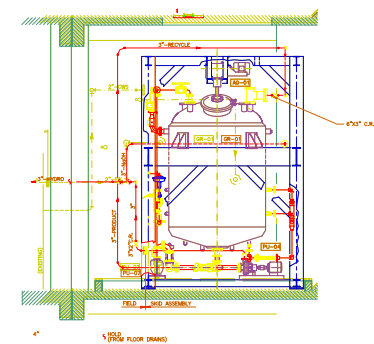
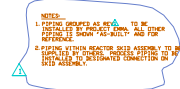
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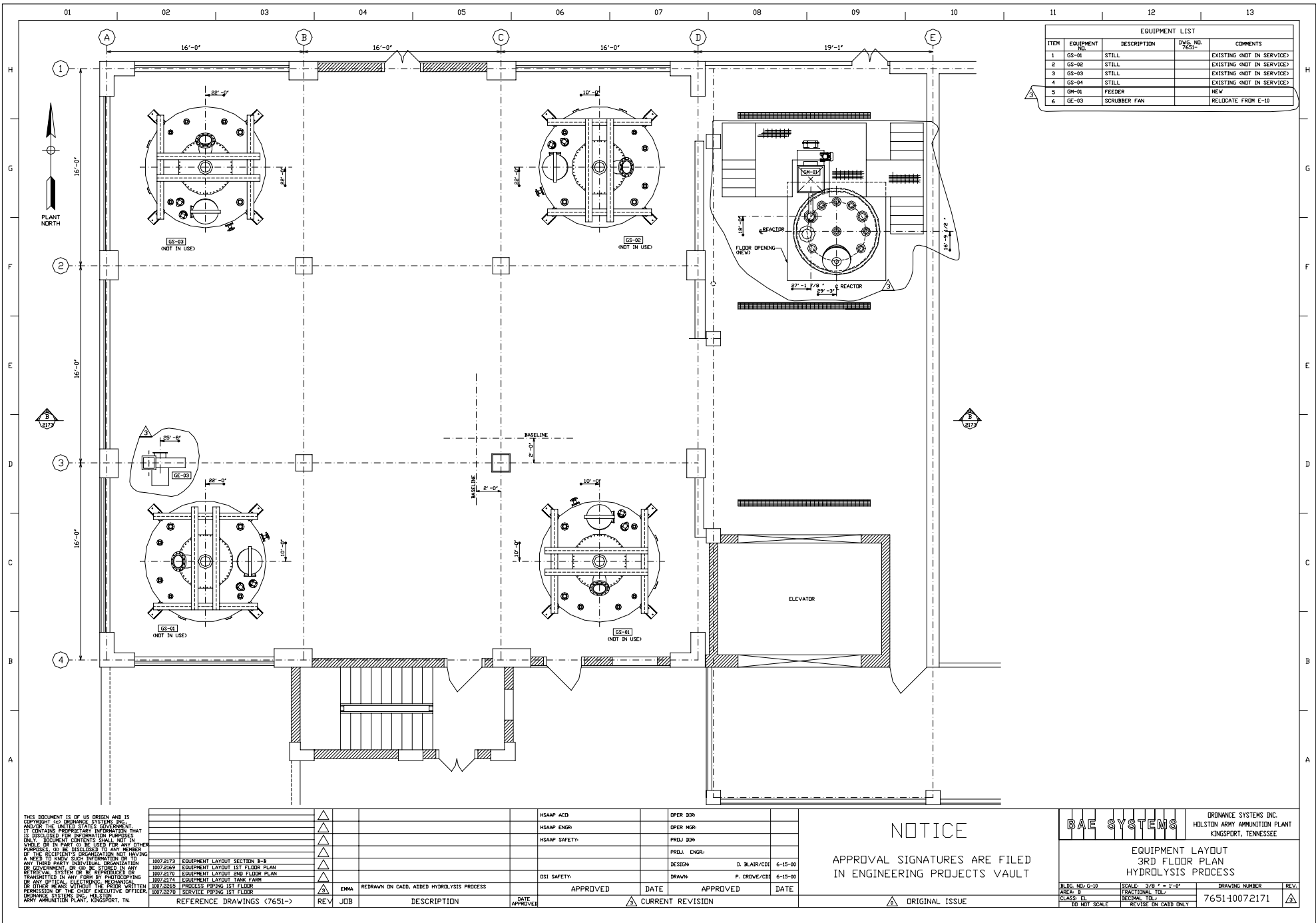
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HSAMP ENGR	OPER MGR
HSAMP SAFETY	PROJ. EDR
DESIGN	S. BLAIR/CDB 6-15-00
DRAWN	P. ORNIE/CDB 6-15-00
DATE	DATE

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BAE SYSTEMS		ORDNANCE SYSTEMS INC. HOLSTON ARMY AMMUNITION PLANT KINGSFORD, TENNESSEE	
EQUIPMENT LAYOUT SECTION A-A HYDROLYSIS PROCESS		DRAWING NUMBER	REV
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CLASS: EL		REVISION: TOL	
DO NOT SCALE		REVISE ON CAD ONLY	





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2	GS-02	STILL		EXISTING (NOT IN SERVICE)
3	GS-03	STILL		EXISTING (NOT IN SERVICE)
4	GS-04	STILL		EXISTING (NOT IN SERVICE)
5	GM-01	FEEDER FAN		NEW
6	GE-03	SCRUBBER FAN		RELOCATE FROM E-10

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REV	JOB	DESCRIPTION	DATE APPROVED	DATE	APPROVED	DATE	APPROVED	DATE
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2		1007.2169 EQUIPMENT LAYOUT 1ST FLOOR PLAN						
3		1007.2170 EQUIPMENT LAYOUT 2ND FLOOR PLAN						
4		1007.2174 EQUIPMENT LAYOUT TANK FARM						
5		1007.2265 PROCESS PIPING 1ST FLOOR						
6		1007.2278 SERVICE PIPING 1ST FLOOR						

ENR	REDRAWN ON CADD, ADDED HYDROLYSIS PROCESS	APPROVED	DATE	APPROVED	DATE
REV	JOB	DESCRIPTION	DATE APPROVED	DATE	APPROVED

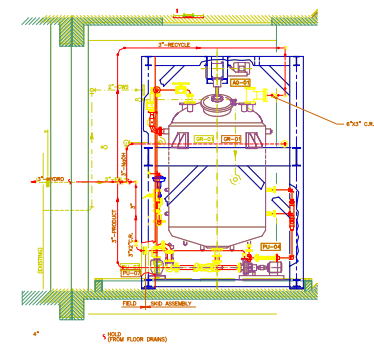
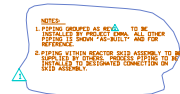
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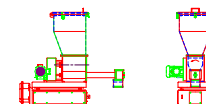
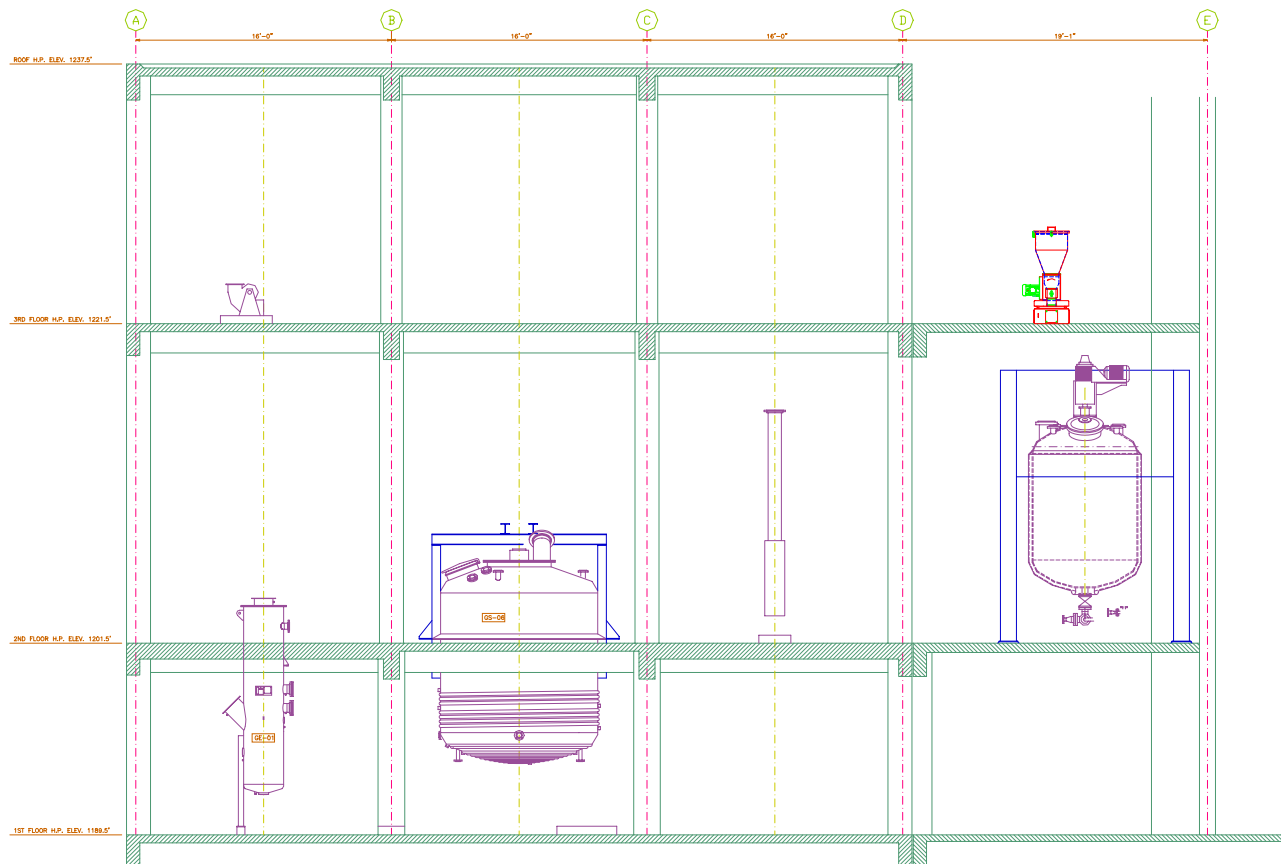
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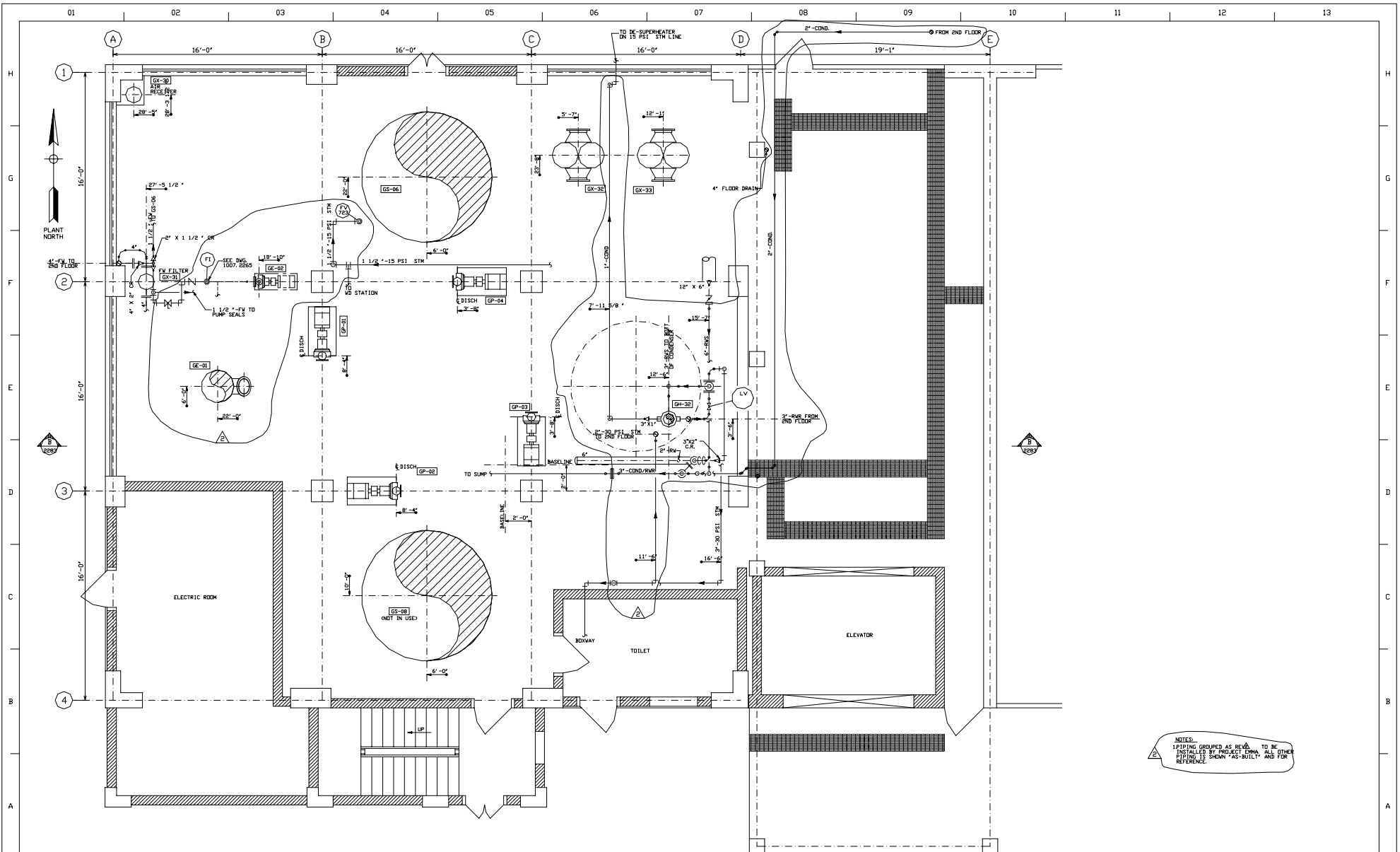
**BAE SYSTEMS**  
DRINANCE SYSTEMS INC.  
HOLLISTON ARMY AMMUNITION PLANT  
KINGSFORD, TENNESSEE

**EQUIPMENT LAYOUT  
3RD FLOOR PLAN  
HYDROLYSIS PROCESS**

BUILDING NO. G-10	SCALE: 3/8" = 1'-0"	DRAWING NUMBER	REV.
AREA: 9	FRACTIONAL TOL.	7651-10072171	
CLASS: EL	DECIMAL TOL.		
DO NOT SCALE	REVISE ON CADD ONLY		







NOTES:  
 1. PIPING GROUPED AS REVISED TO BE  
 2. INSTALLED BY PROJECT TEAM. ALL OTHER  
 3. PIPING IS SHOWN "AS-BUILT" AND FOR  
 4. REFERENCE.

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 STORAGE AND RETRIEVAL SYSTEM, WITHOUT  
 PERMISSION OF THE COMMANDING OFFICER,  
 DRONANCE SYSTEMS INC., HOLSTON ARMY  
 AMMUNITION PLANT, KINGSFORD, TN.

1007.2279	SERVICE PIPING 2ND FLOOR PLAN
1007.2280	SERVICE PIPING 3RD FLOOR PLAN
1007.2281	SERVICE PIPING TANK FARM PLAN
1007.2282	SERVICE PIPING SECTION B-B
1007.2270	EQUIPMENT LAYOUT 2ND FLOOR PLAN
1007.2171	EQUIPMENT LAYOUT 3RD FLOOR PLAN
1007.2265	PROCESS PIPING 1ST FLOOR PLAN
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REV	JOB	DESCRIPTION
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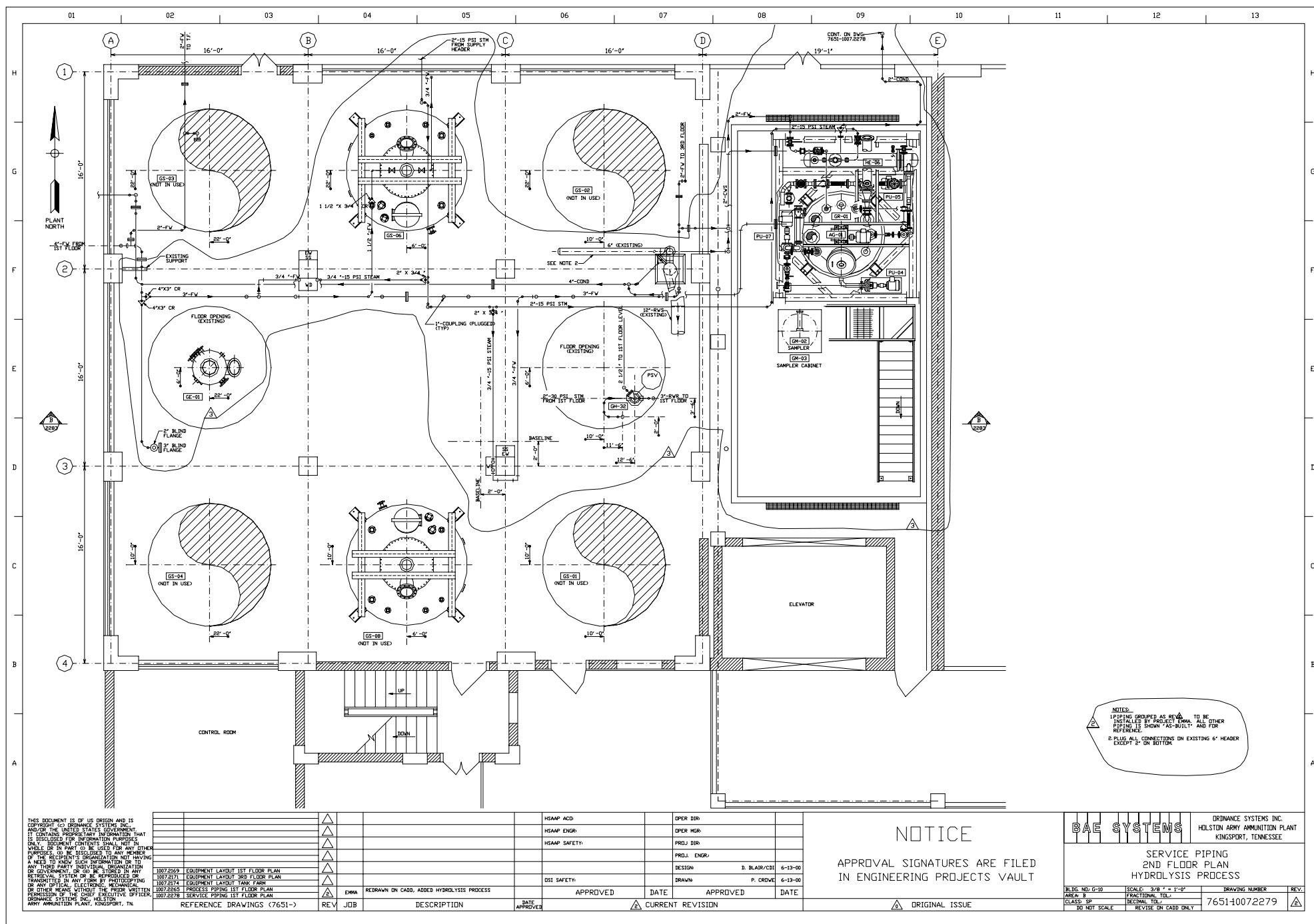
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	APPROVED	DATE	APPROVED	DATE
DATE APPROVED	CURRENT REVISION			

**NOTICE**

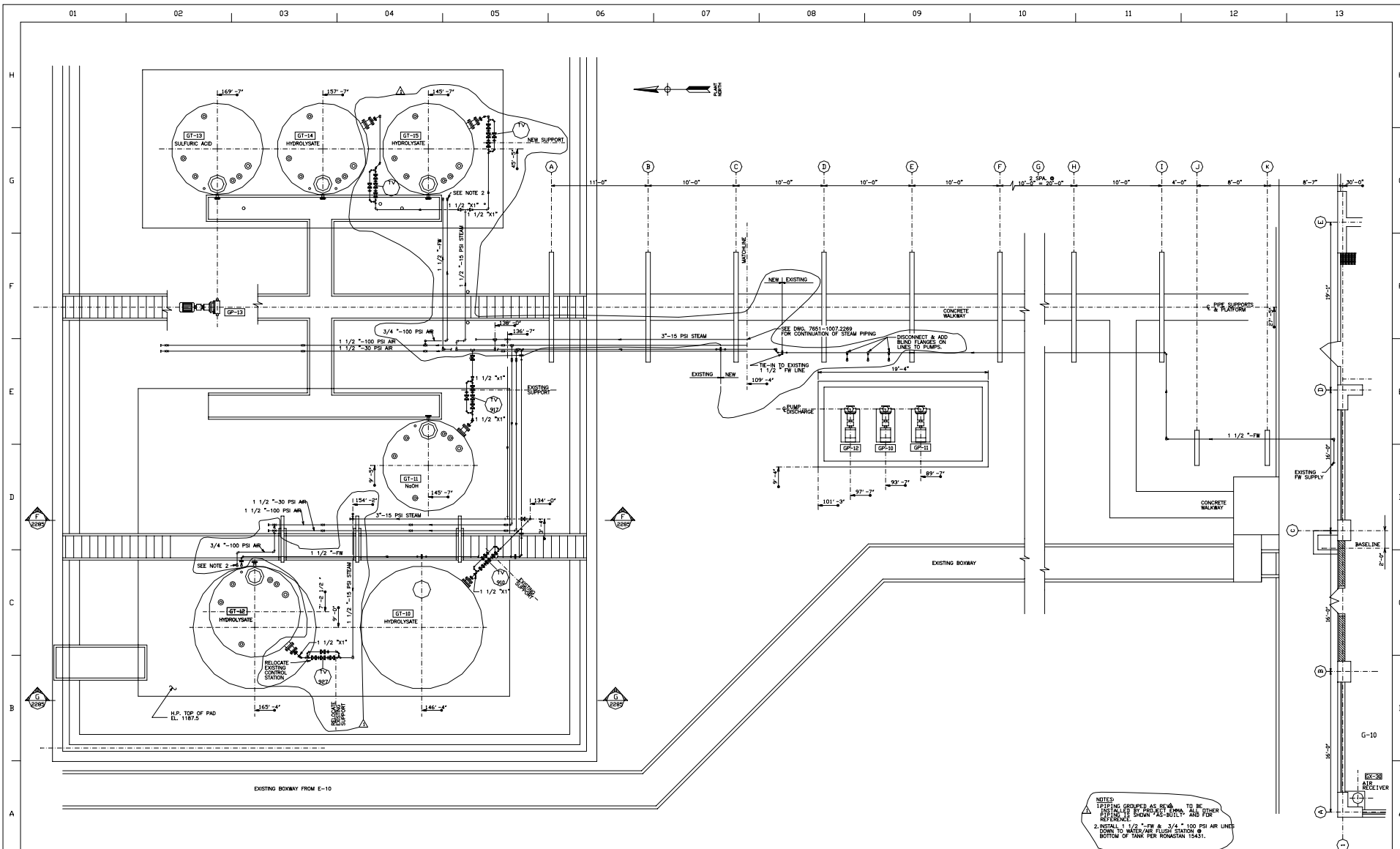
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ORIGINAL ISSUE

BAE SYSTEMS		DRONANCE SYSTEMS INC. HOLSTON ARMY AMMUNITION PLANT KINGSFORD, TENNESSEE	
SERVICE PIPING 1ST FLOOR PLAN HYDROLYSIS PROCESS			
BLDG NO: G-10	SCALE: 3/8" = 1'-0"	DRAWING NUMBER	REV.
AREA: B	FRACTIONAL TOL:	7651-1007.2278	
CLASS: SP	DECIMAL TOL:		
NO. NOT SCALE	REVISE ON CAB ONLY		







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10072595	TANK FARM LAYOUT & SECTION
10072596	PROCESS PIPING TANK FARM PLAN
10072597	EQUIPMENT LAYOUT TANK FARM PLAN
10072598	EQUIPMENT LAYOUT 2ND FLOOR PLAN
10072599	EQUIPMENT LAYOUT 3RD FLOOR PLAN
10072600	PROCESS PIPING 1ST FLOOR PLAN
10072601	SERVICE PIPING 1ST FLOOR PLAN

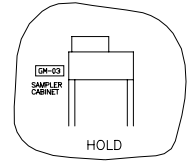
ENR	REDESIGN ON CASE, ADDS HYDROLYSIS PROCESS
REV	JOB
DESCRIPTION	
DATE	APPROVED

HSAMP ACID	OPER DIR:
HSAMP ENGR:	OPER MGR:
HSAMP SAFETY:	PROJ DIR:
	PROJ ENGR:
	DESIGN
	DRAWN


DATE	APPROVED	DATE	APPROVED
DATE	APPROVED	DATE	APPROVED

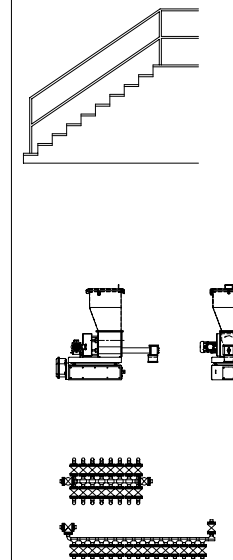
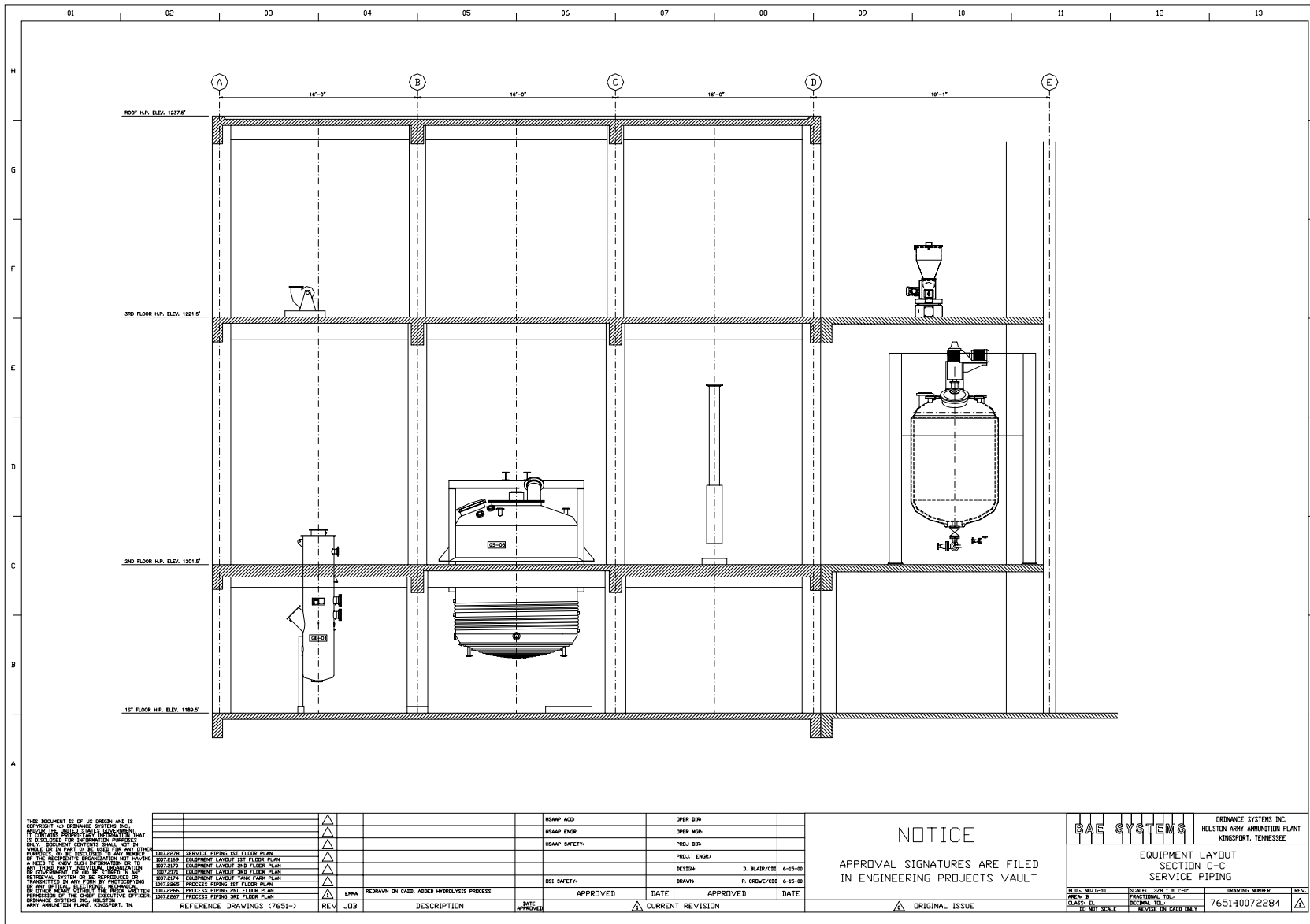
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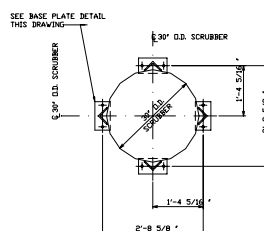
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SERVICE PIPING TANK FARM PLAN HYDROLYSIS PROCESS			
BLDG NO. G-10	SCALE 1/4" = 1'-0"	DRAWING NUMBER	REV.
AREA B	FRACTIONAL TOL.	7651-1007.2281	
CLASS 3P	DECIMAL TOL.		
3D NET SCALE	REVISE ON CASE ONLY		



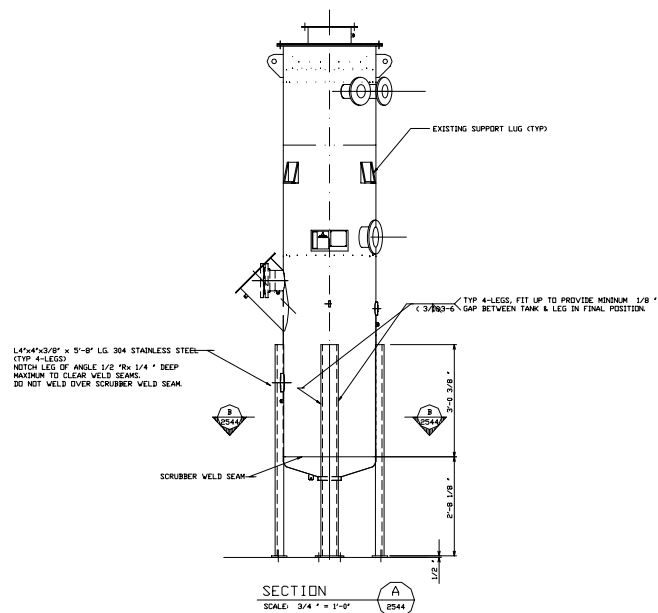
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SERVICE PIPING SECTION B-B HYDROLYSIS PROCESS			
BLDG. NO.: G-10	SCALE: 3/8" = 1'-0"	DRAWING NUMBER	RE
AREA	FRACTIONAL TOL.	7651-10072283	A
CLASS: SP	DECIMAL TOL.		

BUILDG. NO: G-10	SCALE: 3/8" = 1'-0"	DRAWING NUMBER	RE
AREA: B	FRACTIONAL TOL.	7651-10072283	
CLASS: SP	DECIMAL TOL.		
DO NOT SCALE		DESIGN ON CARD ONLY	





SECTION B  
SCALE: 3/4" = 1'-0" 2544



BASE PLATE DETAIL- 4 REQ'D  
SCALE: 3" = 1'-0"

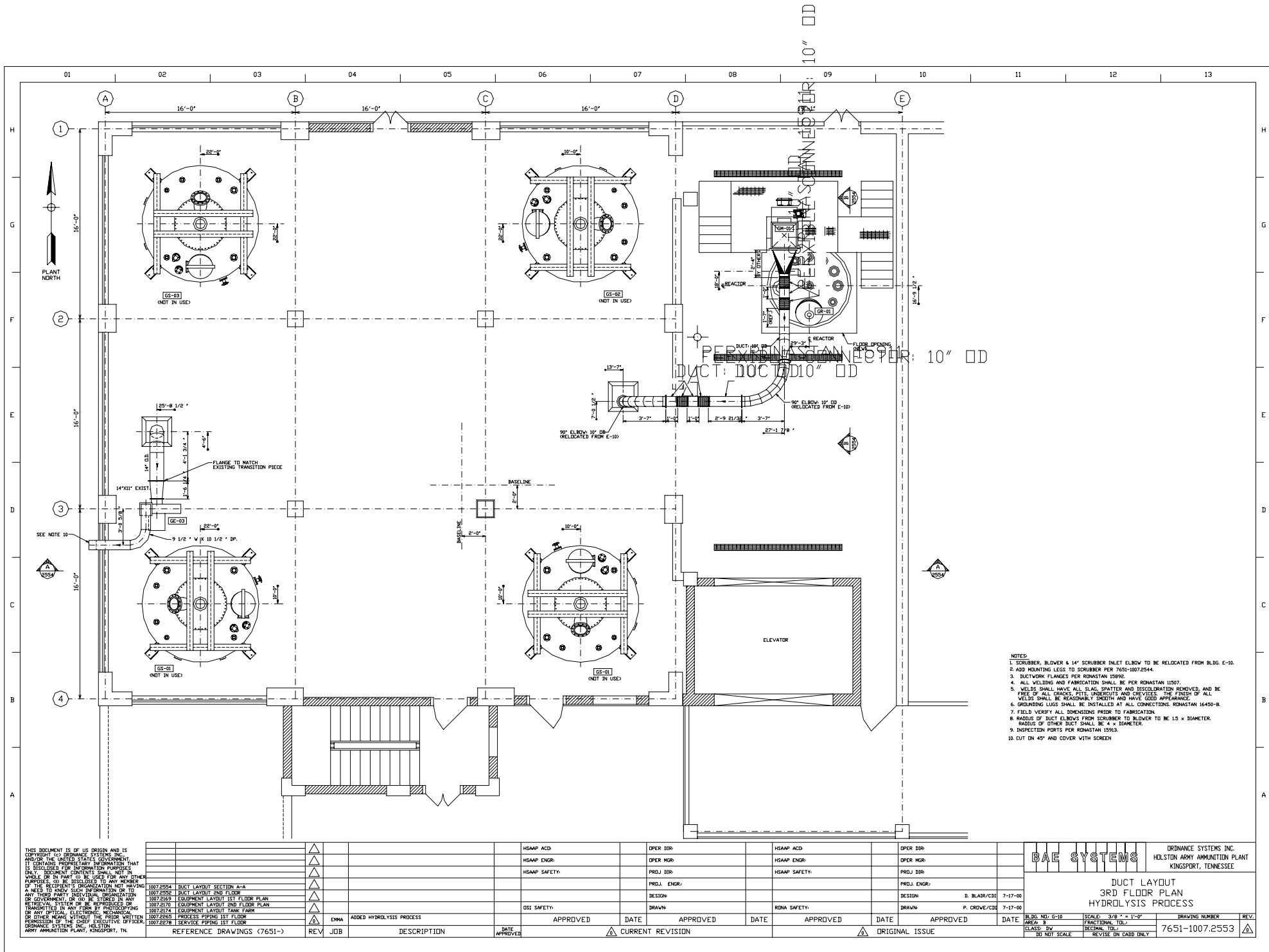
NOTES:

1. FOR GENERAL NOTES SEE DRAWING 7651-1009.1614
2. SEE 7651-1009.0634 FOR ORIGINAL SCRUBBER DRAWING.

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ORDNANCE SYSTEMS INC. HILLSTO  
ARMY AMMUNITION PLANT, KINGSPORT, TN.

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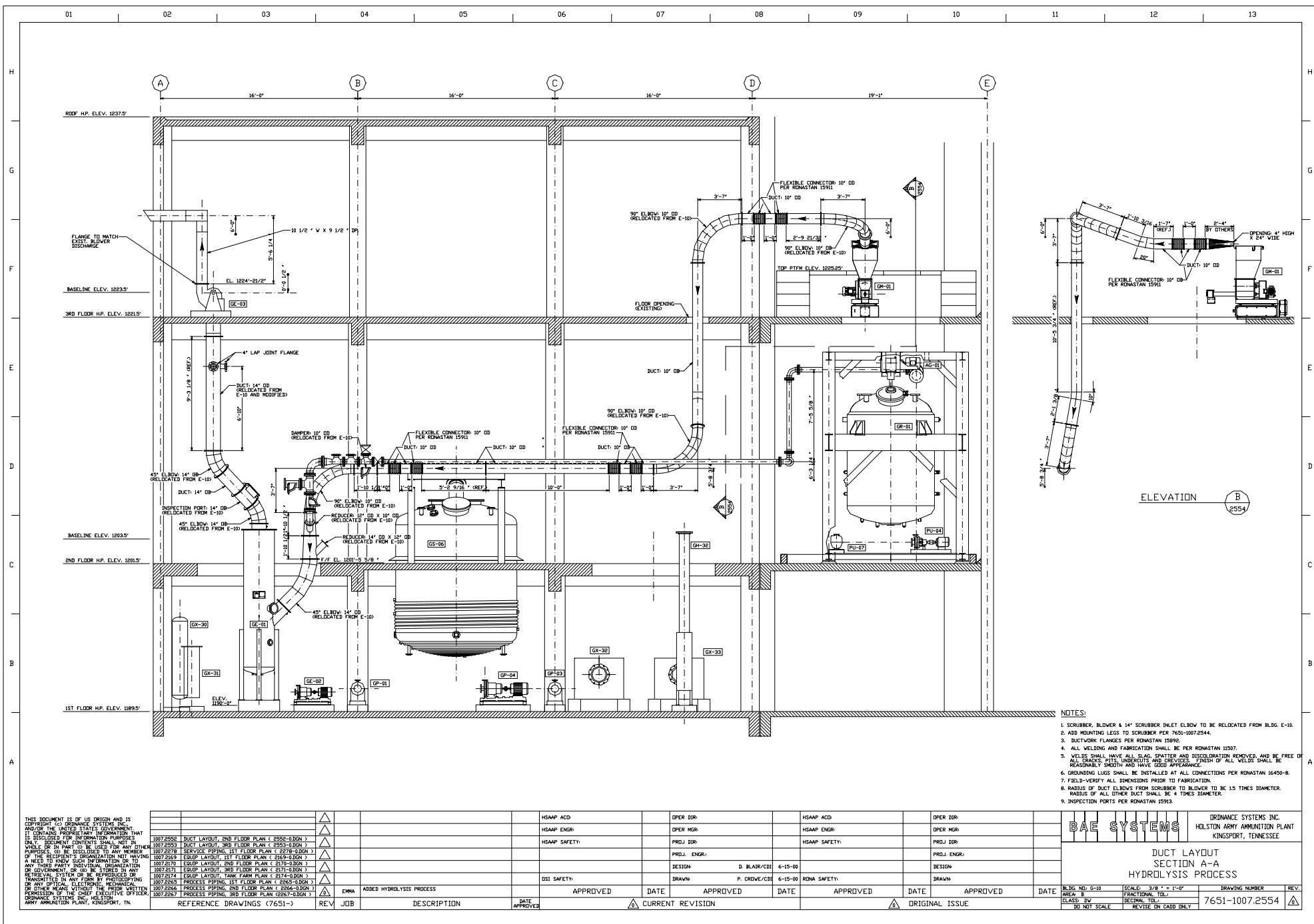


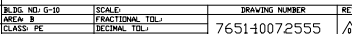


- NOTES:
1. SCRUBBER, BLOWER & 14" SCRUBBER INLET ELBOW TO BE RELOCATED FROM BLDG. E-10.
  2. ADD MOUNTING LEGS TO SCRUBBER PER 7651-1007.2544.
  3. DUCTWORK FLANGES PER ROMANSTAN 15590.
  4. ALL WELDING AND FABRICATION SHALL BE PER ROMANSTAN 11507.
  5. WELDS SHALL HAVE ALL SLAG, SPATTER AND DISCOLORATION REMOVED, AND BE FREE OF ALL CRACKS, FITS, UNDERCUTS AND CRACKS. THE FINISH OF ALL WELDS SHALL BE REASONABLY SMOOTH AND HAVE GOOD APPEARANCE.
  6. GROUNDING LUGS SHALL BE INSTALLED AT ALL CONNECTIONS. ROMANSTAN 16450-B.
  7. FIELD VERIFY ALL DIMENSIONS PRIOR TO FABRICATION.
  8. RADIUS OF DUCT ELBOWS FROM SCRUBBER TO BLOWER TO BE 15 x DIAMETER. RADIUS OF OTHER DUCT SHALL BE 4 x DIAMETER.
  9. INSPECTION PORTS PER ROMANSTAN 15593.
  10. CUT ON 45° AND COVER WITH SCREEN.

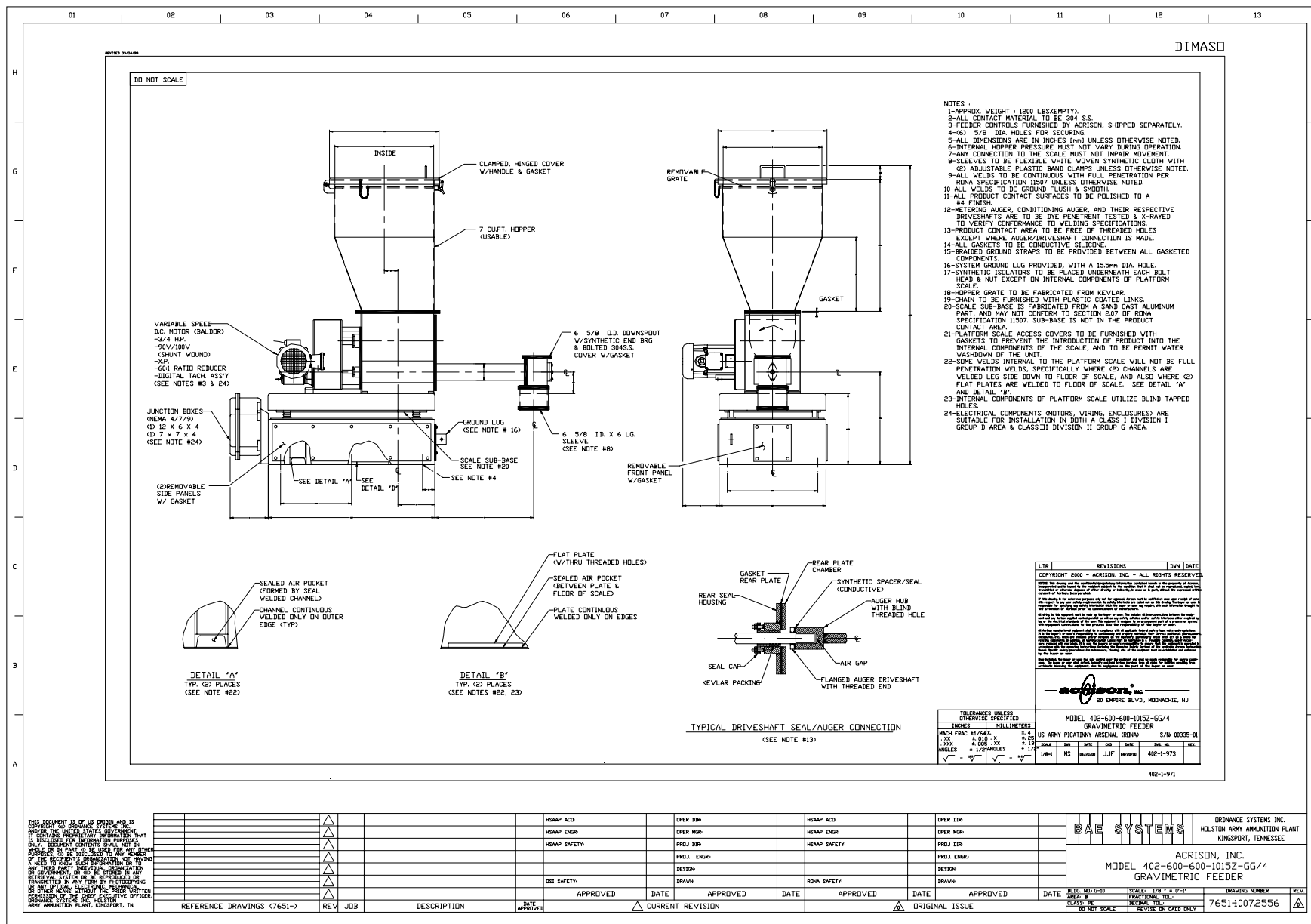
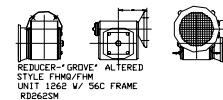
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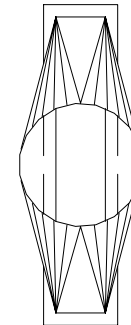
		▲			HSAMP ACQ		OPER DIR		HSAMP ACQ		OPER DIR	BAE SYSTEMS	ORDNANCE SYSTEMS INC. HOLSTON ARMY AMMUNITION PLANT KINGSPORT, TENNESSEE	
		▲			HSAMP ENGR		OPER MGR		HSAMP ENGR		OPER MGR			
		▲			HSAMP SAFETY		PROJ DIR		HSAMP SAFETY		PROJ DIR			
1007.2554		▲					PROJ. ENGR				PROJ. ENGR	DUCT LAYOUT 3RD FLOOR PLAN HYDROLYSIS PROCESS		
1007.2555		▲					DESIGN				DESIGN			
1007.2556		▲					OSI SAFETY				RONA SAFETY			
1007.2570		▲					DRAWN				DRAWN	7-17-00		
1007.2574		▲										7-17-00		
1007.2565		▲												
1007.2578		▲												
REFERENCE DRAWINGS (7651-)		REV	JOB	DESCRIPTION	DATE	APPROVED	DATE	APPROVED	DATE	APPROVED	DATE	BLDG. NO: G-10		
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												REV.		





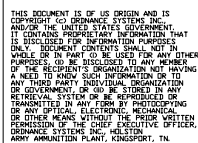




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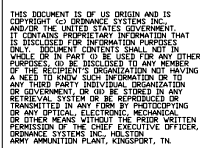
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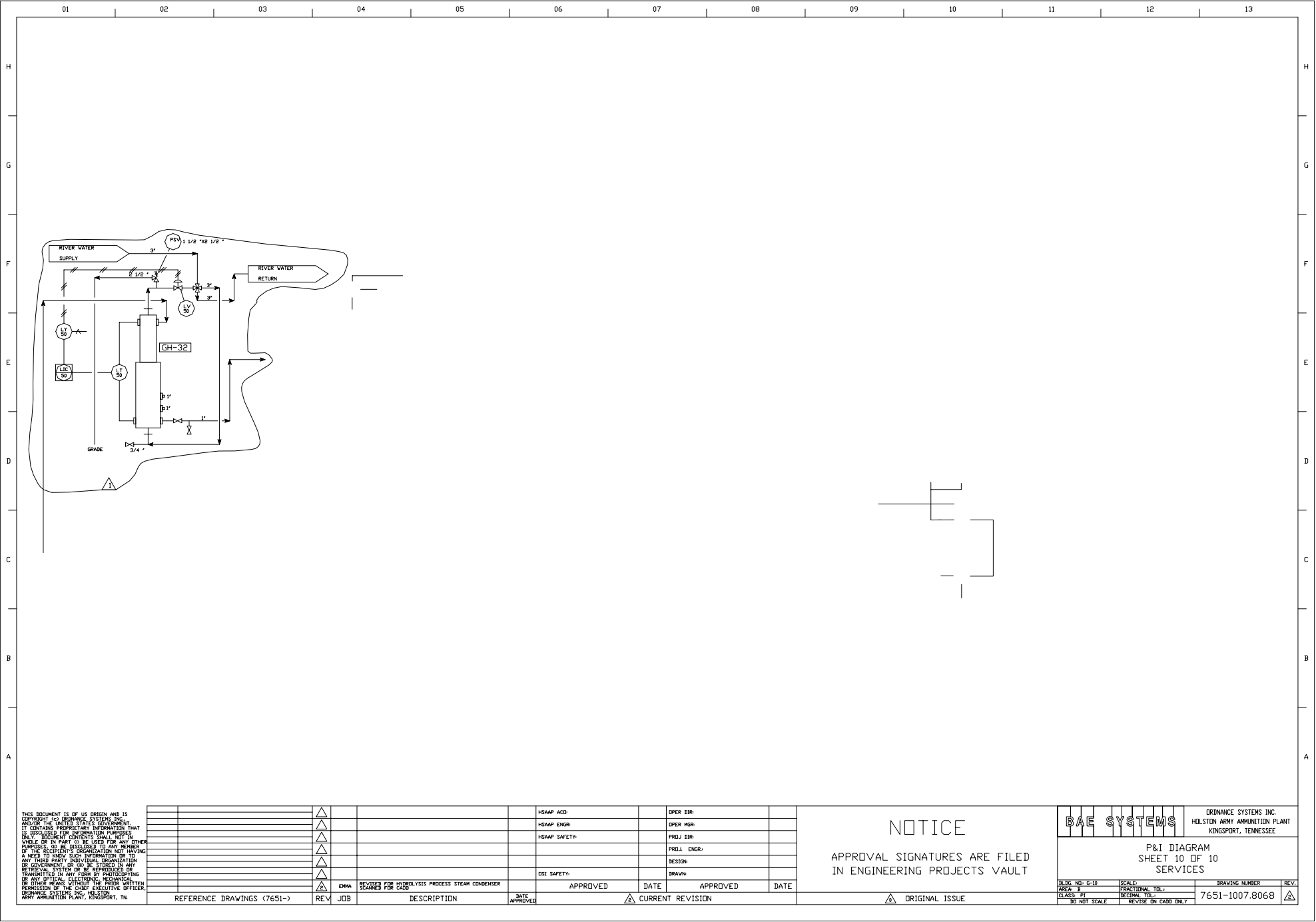
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REV	JOB	DESCRIPTION	DATE APPROVED	APPROVED	DATE	APPROVED	DATE
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				HSAMP ENGR:		OPER MGR:	
				HSAMP SAFETY:		PRJL DIR:	
						PRJL ENGR:	
						DESIGN:	
				DSI SAFETY:		DRAWN:	
				ENHA	DESIGNED FOR HYDROLYSIS PROCESS STEAM CONDENSER		
					EXAMINED FOR CAD		

NOTICE

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BAE SYSTEMS

DRINANCE SYSTEMS INC.  
HOLSTON ARMY AMMUNITION PLANT  
KINGSFORT, TENNESSEE

P&I DIAGRAM  
SHEET 10 OF 10  
SERVICES

BLDG. NO.: G-10  
AREA: 8

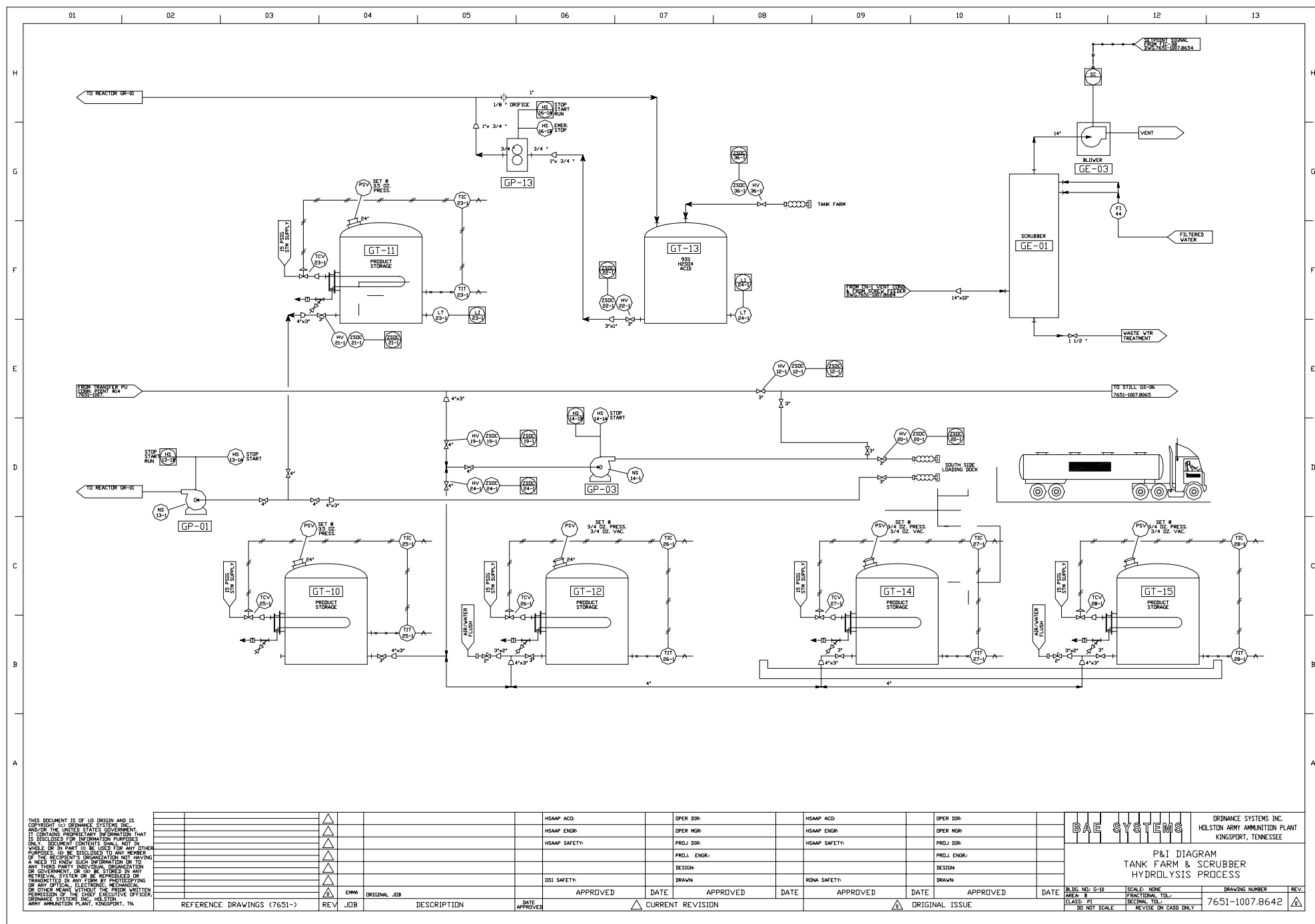
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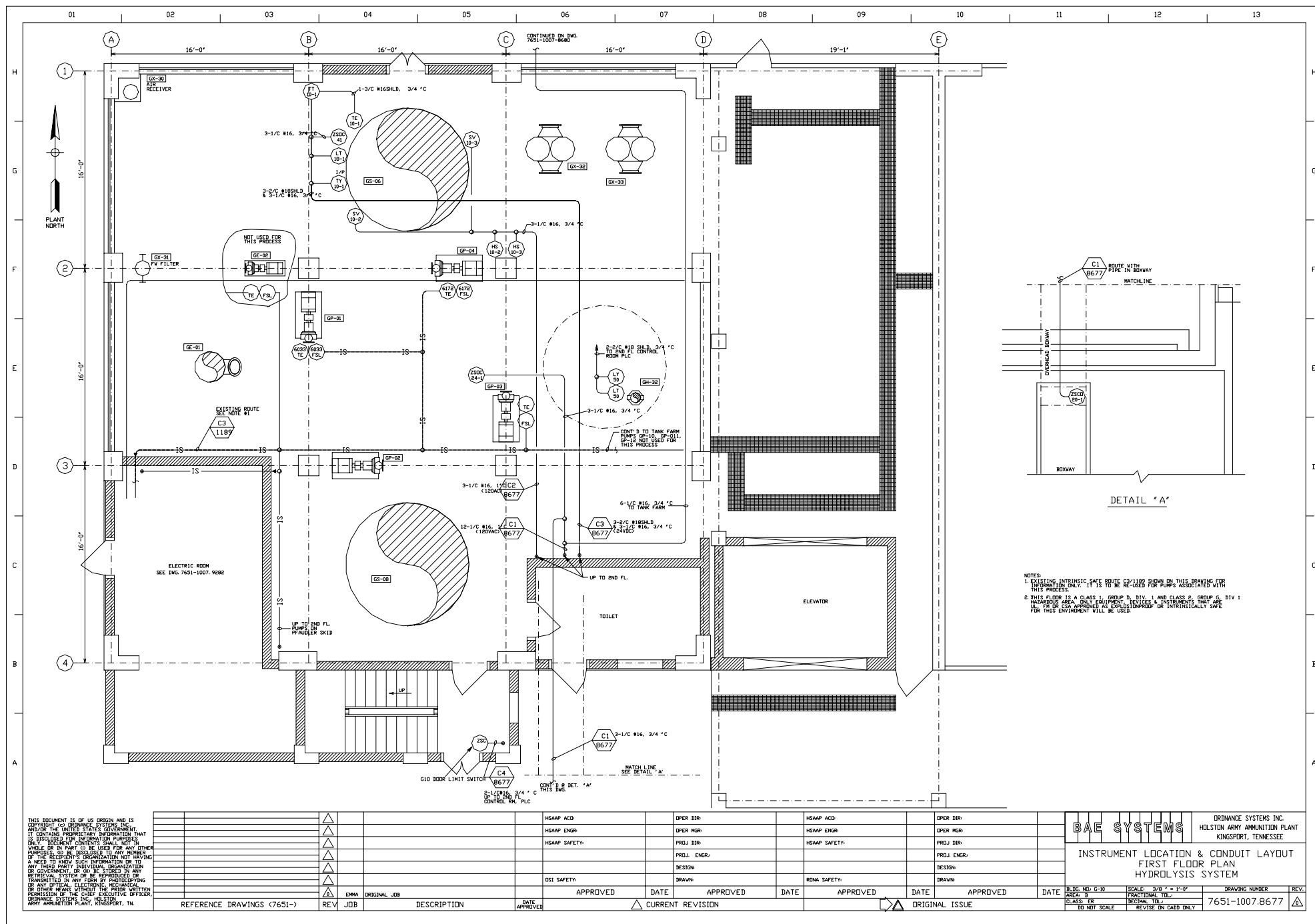
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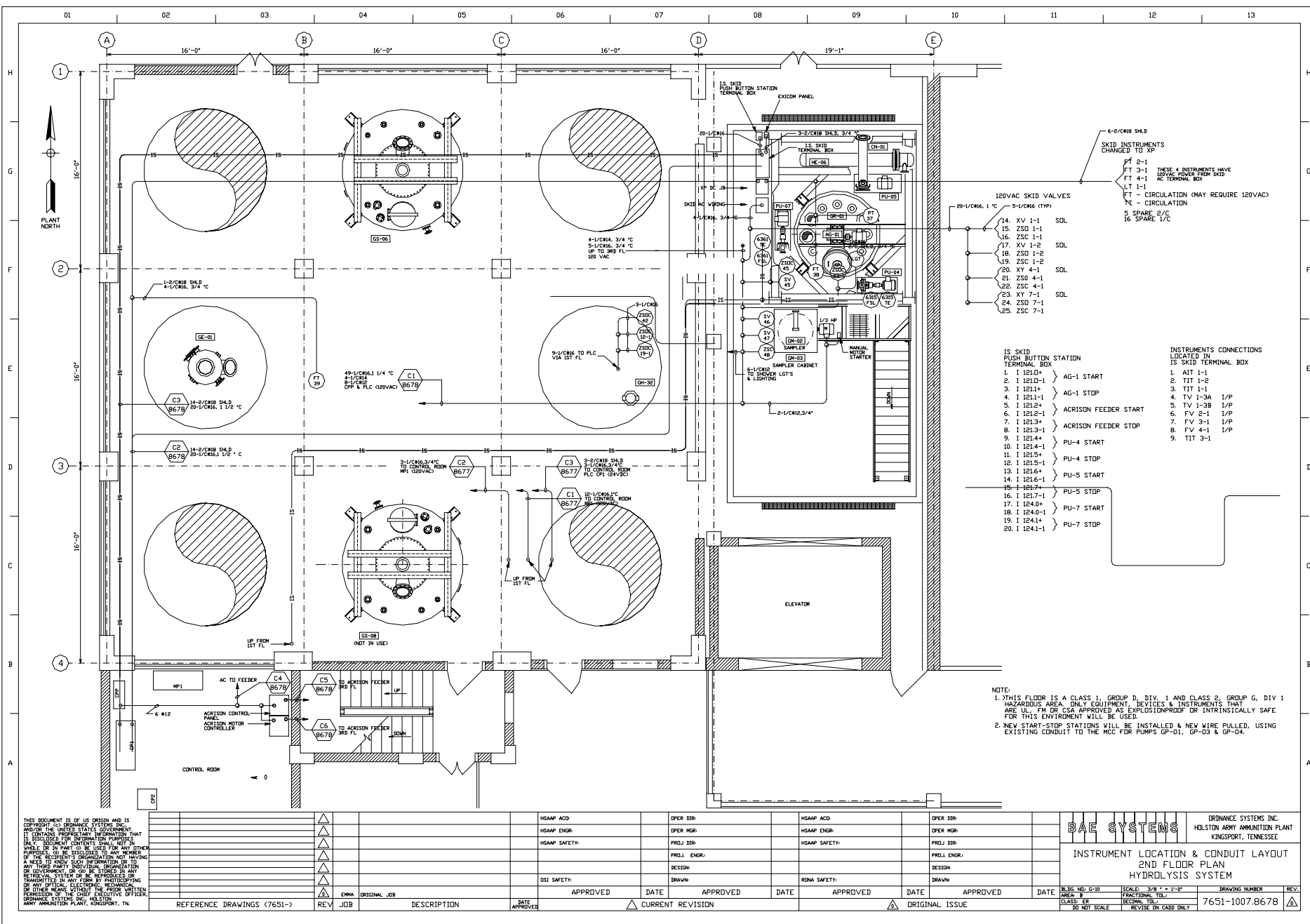
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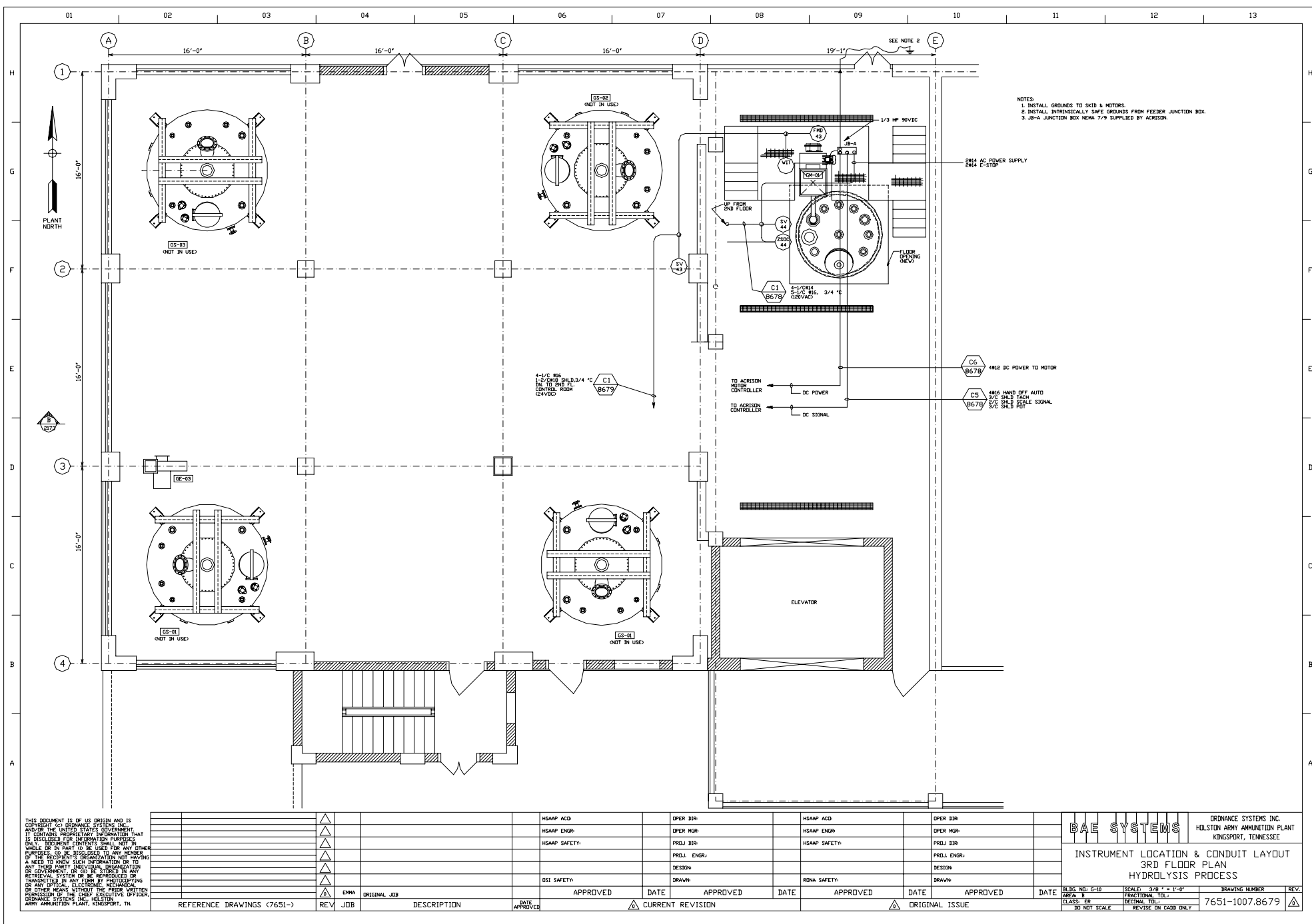


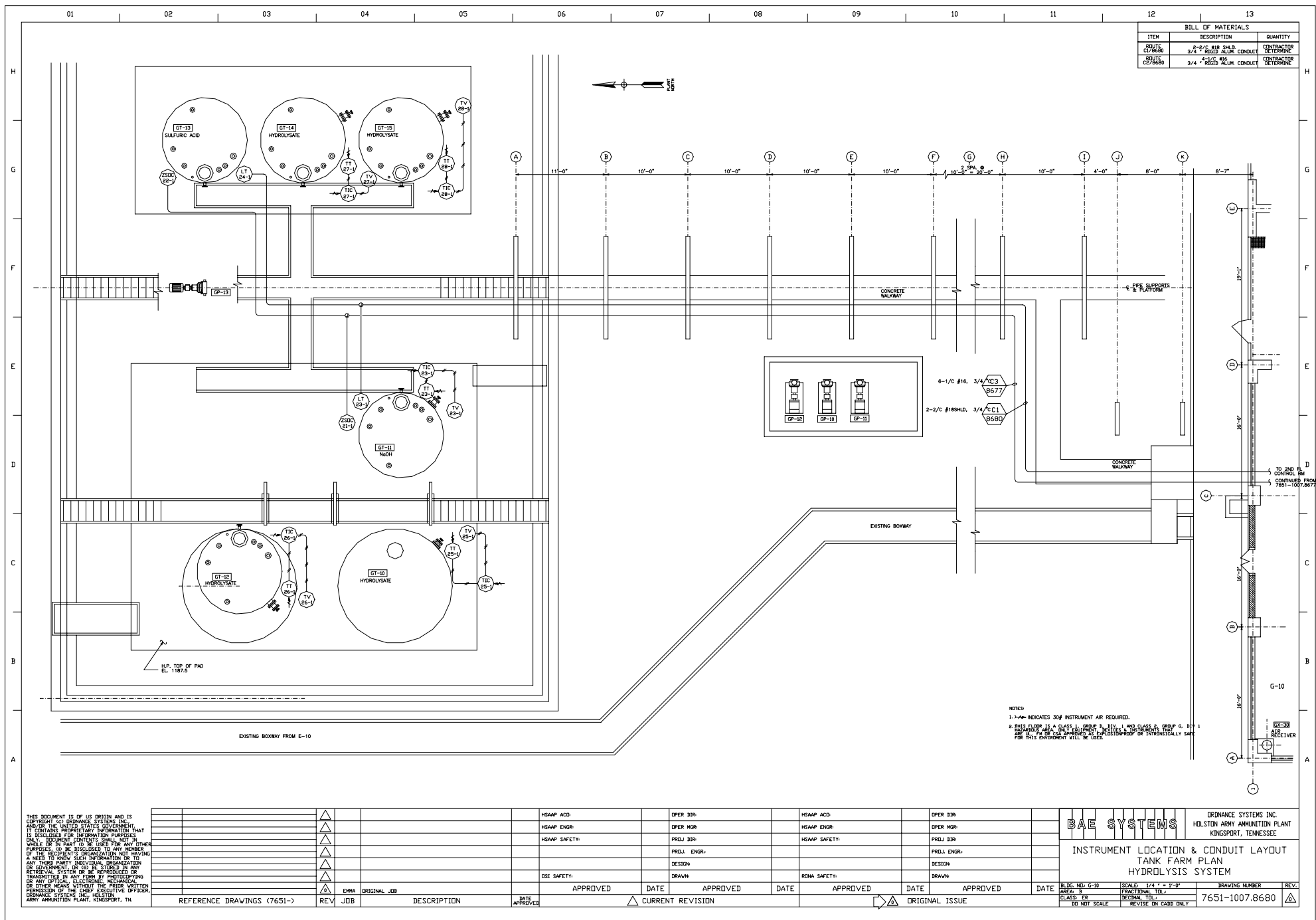


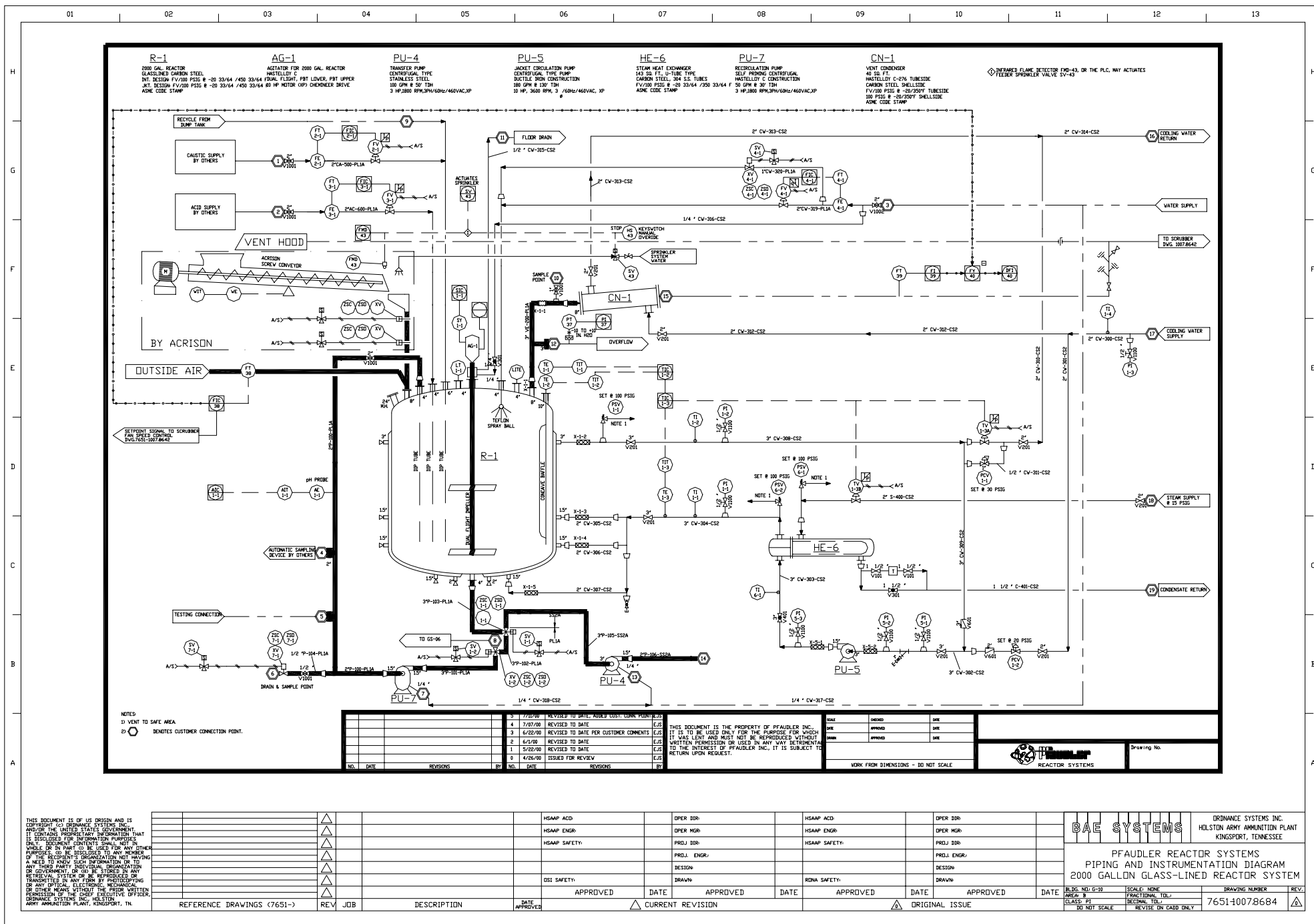


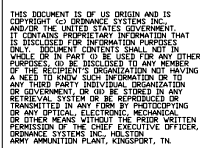


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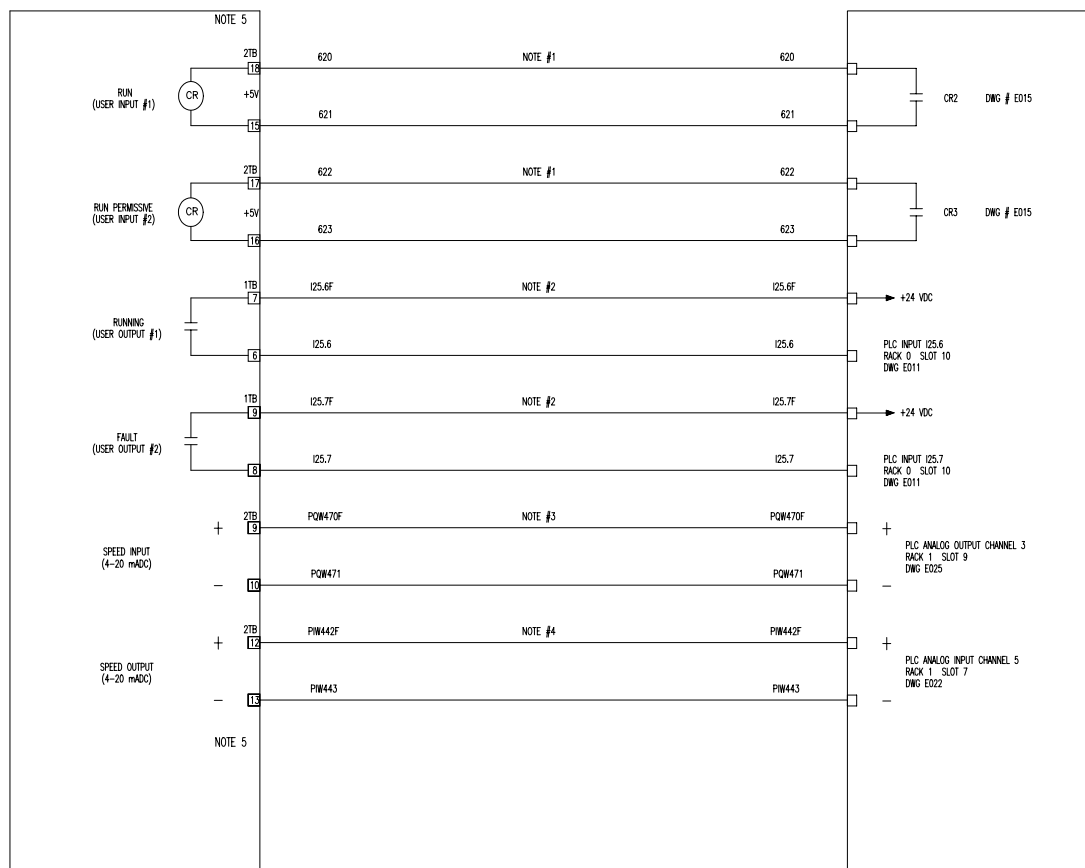
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EMMA ELEMENTARY DIAGRAM CONTROL SYS. REACTOR AGITATOR WIRING HYDROLYSIS SYSTEM			
BLOC. NO. G-10	SCALE NONE	DRAWING NUMBER	REV.
E	1/FRACTIONAL, TOL. NONE		
CLASS ES	DECIMAL, TOL. NONE	7651-1007.8687	⚠
CLASS ES	SCALE NONE		



## ACRISON CONTROLLER

## PLC PANEL



- NOTES:
- 1) ACRISON SUPPLIES POWER FOR IT'S INPUTS.
  - 2) PLC EXPECTS DRY CONTACTS FOR ACRISON DIGITAL OUTPUTS.
  - 3) PLC HAS ACTIVE TRANSMITTER FOR 4-20 mA DC SIGNAL TO ACRISON.
  - 4) PLC EXPECTS ACTIVE TRANSMITTER FOR 4-20 mA DC SIGNAL FROM ACRISON.
  - 5) SEE ACRISON DRAWINGS FOR TERMINAL STRIP NUMBERS.

[illegible]

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SCALE	NONE	CHECKED	DATE
DATE	8/2000	APPROVED	DATE
DRAWN	ROSS	APPROVED	DATE
WORK FROM DIMENSIONS - DO NOT SCALE			

	CONTROL SYSTEM
	ACRISON FEEDER INTERFACE WIRING

**PRIMMER, Inc.**  
A Unit of Robbins & Myers, Inc.

Drawing No.	
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E0005

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ARMY AMMUNITION PLANT, KINGSPORT, TN.

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H N <sub>2</sub>	
	REFERENCE DRAWINGS (7651-)

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REV	JOB	DESCRIPTION

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DATE	

CURRENT REV

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TESTING	

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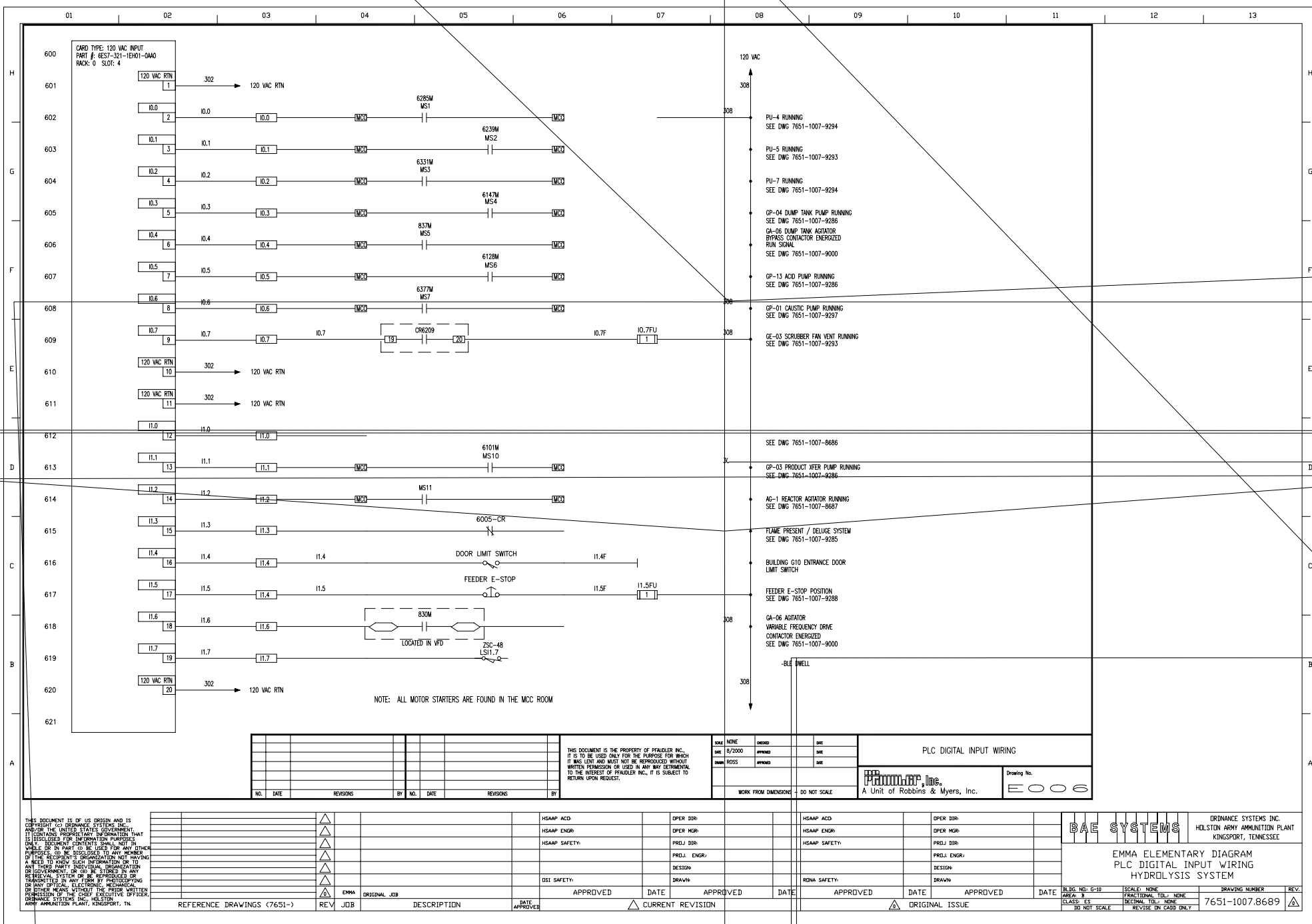
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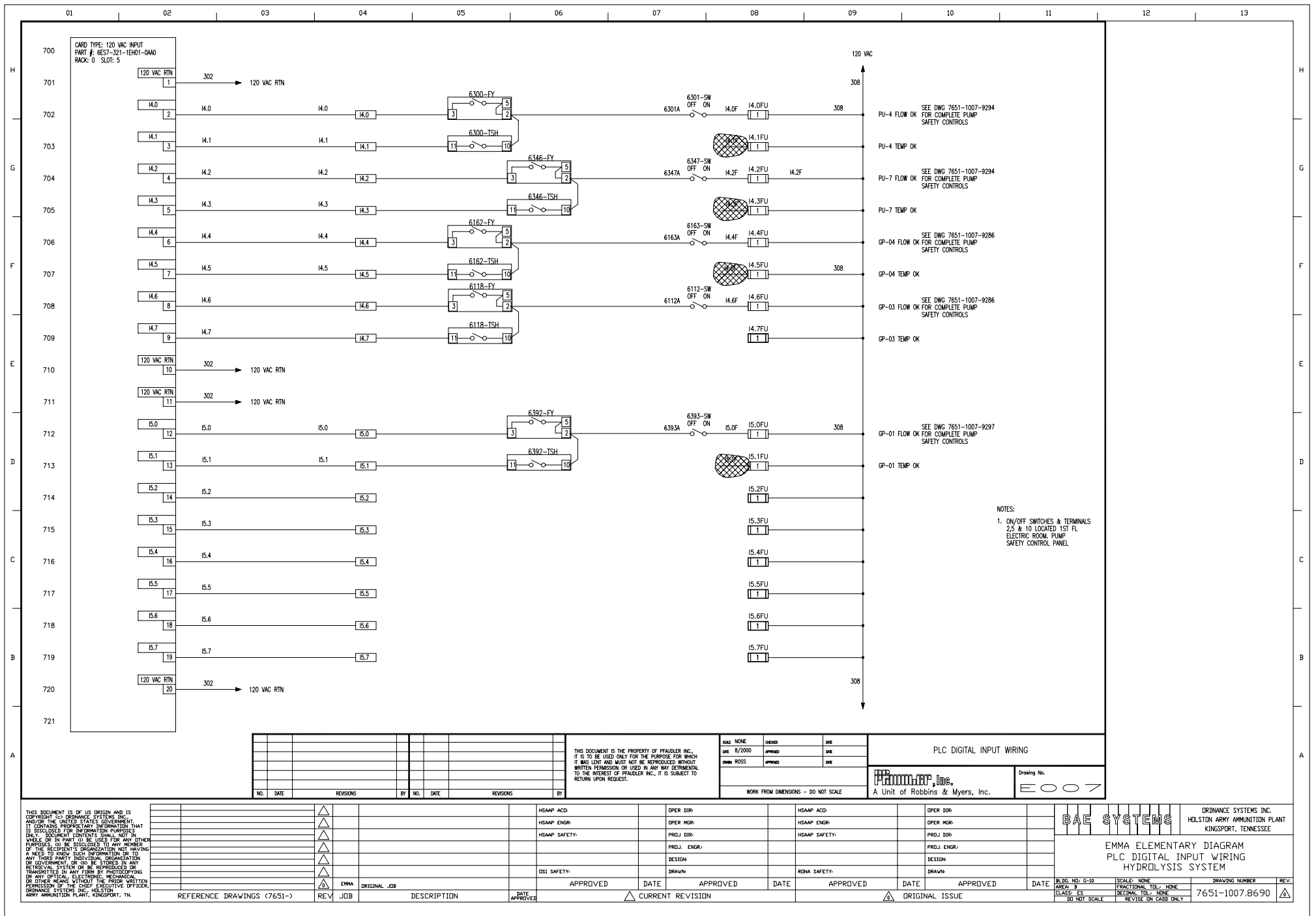
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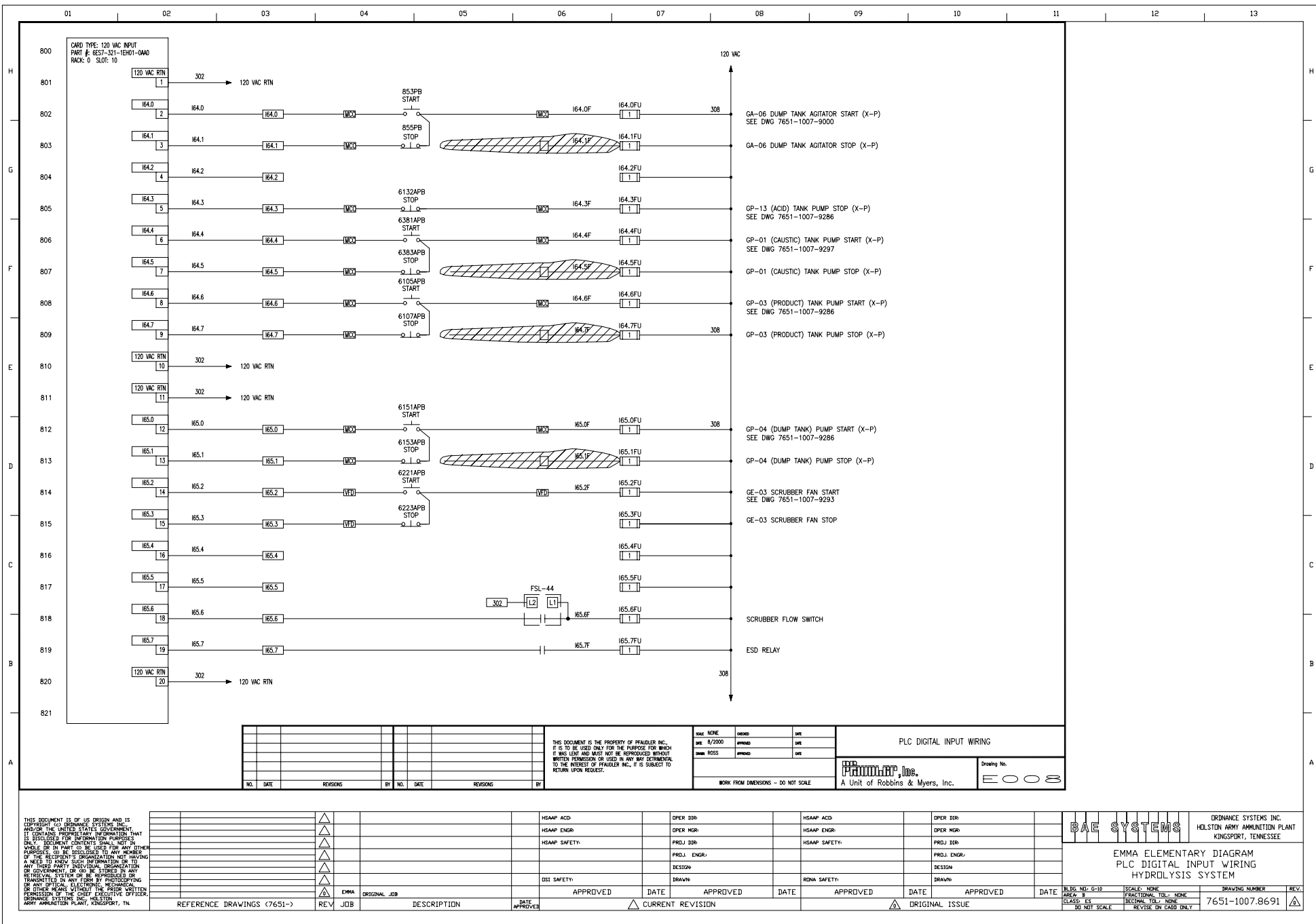
BAE SYSTEMS	ORDNANCE SYSTEMS INC. HOLSTON ARMY AMMUNITION PLANT KINGSPORT, TENNESSEE
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EMMA ELEMENTARY DIAGRAM  
ACRISON FEEDER INTERFACE WIRING  
HYDROLYSIS SYSTEM

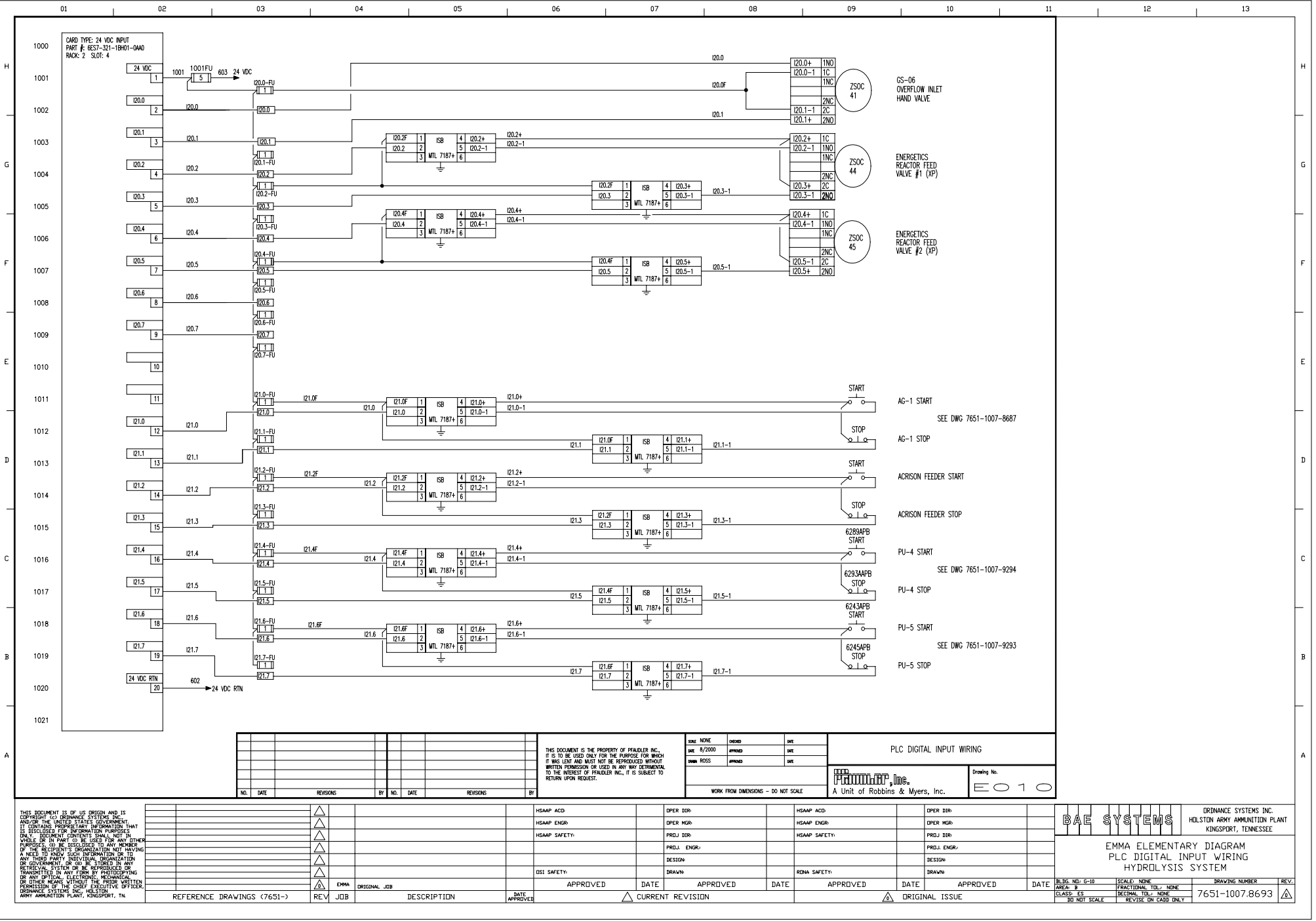
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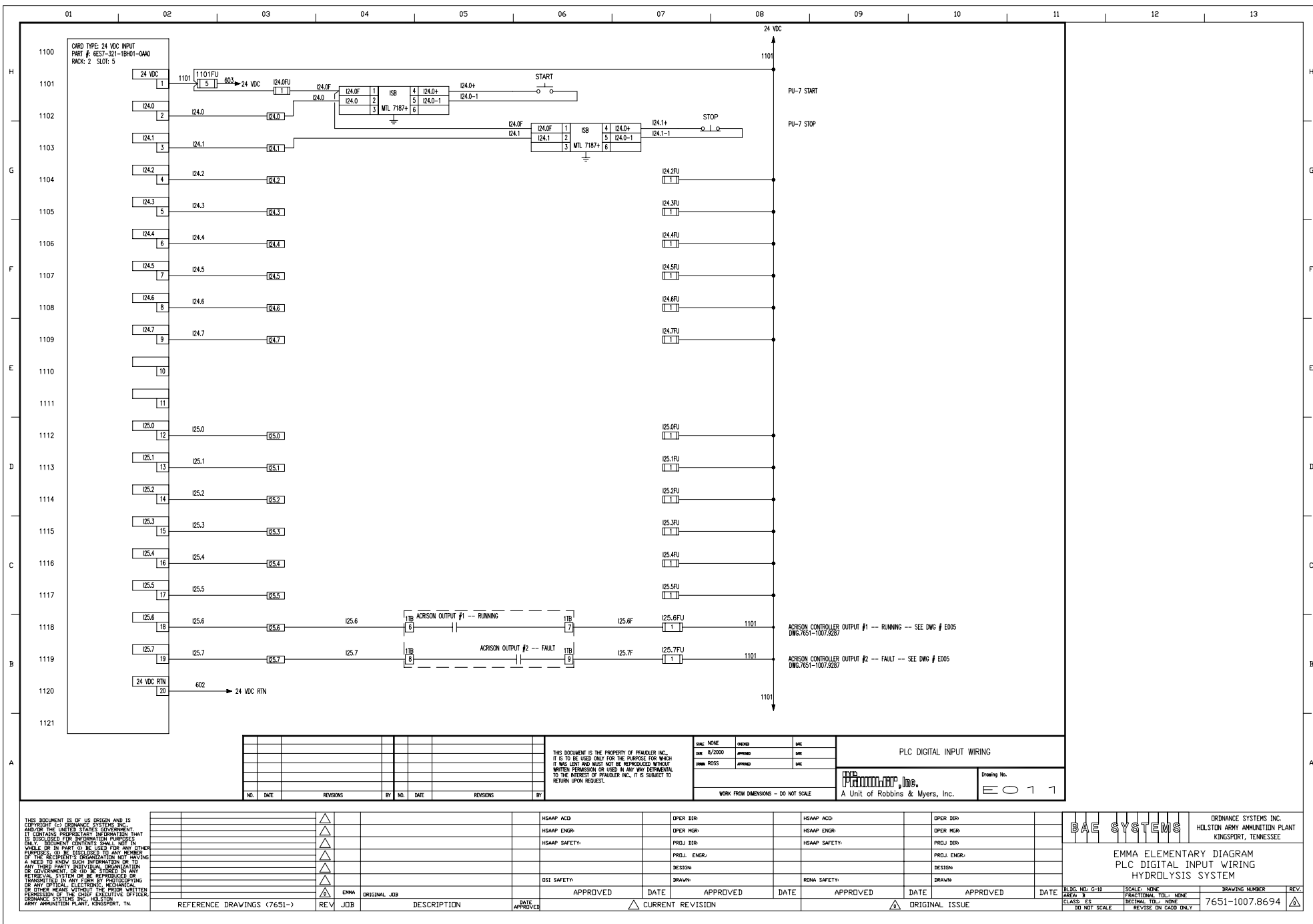


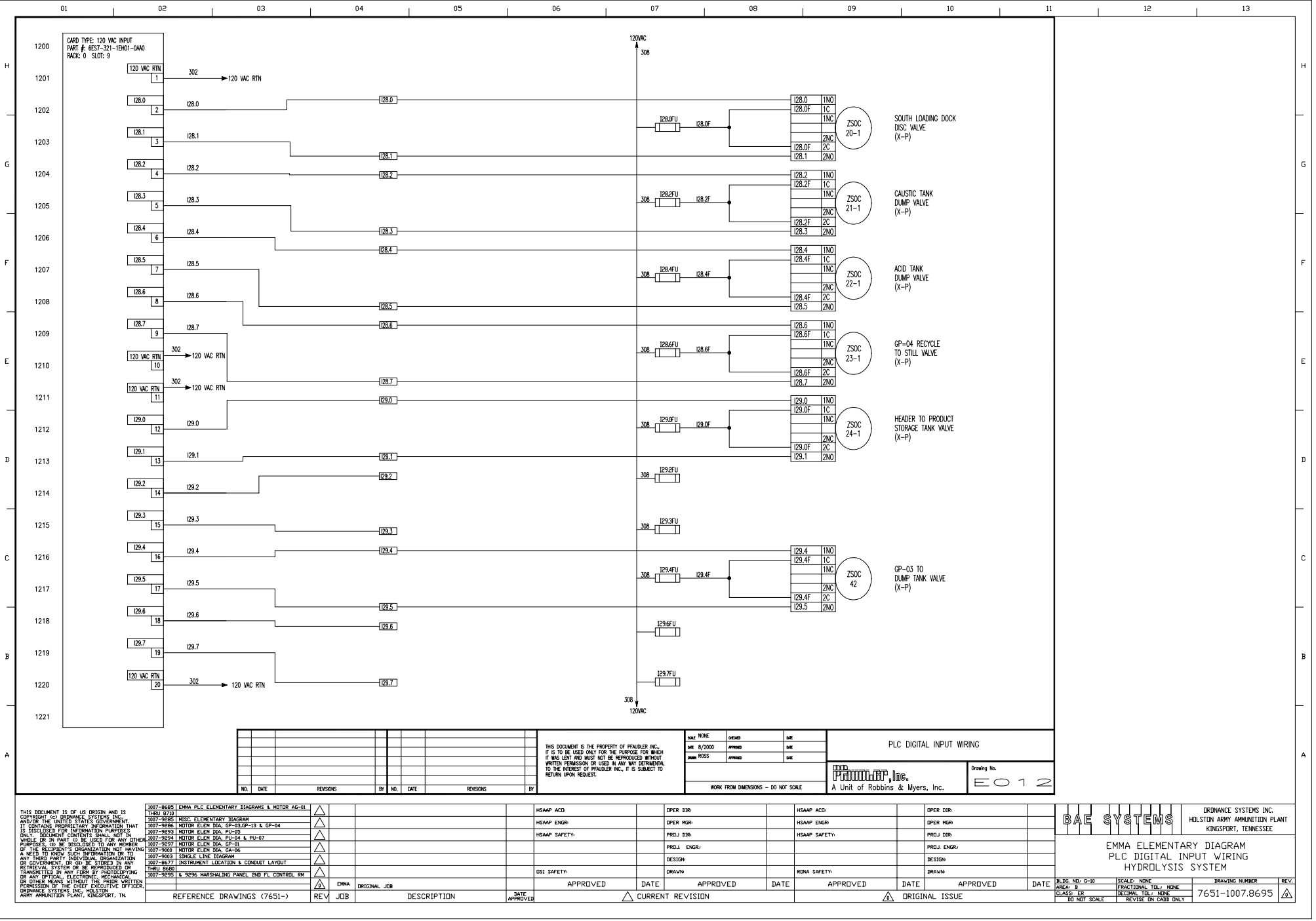




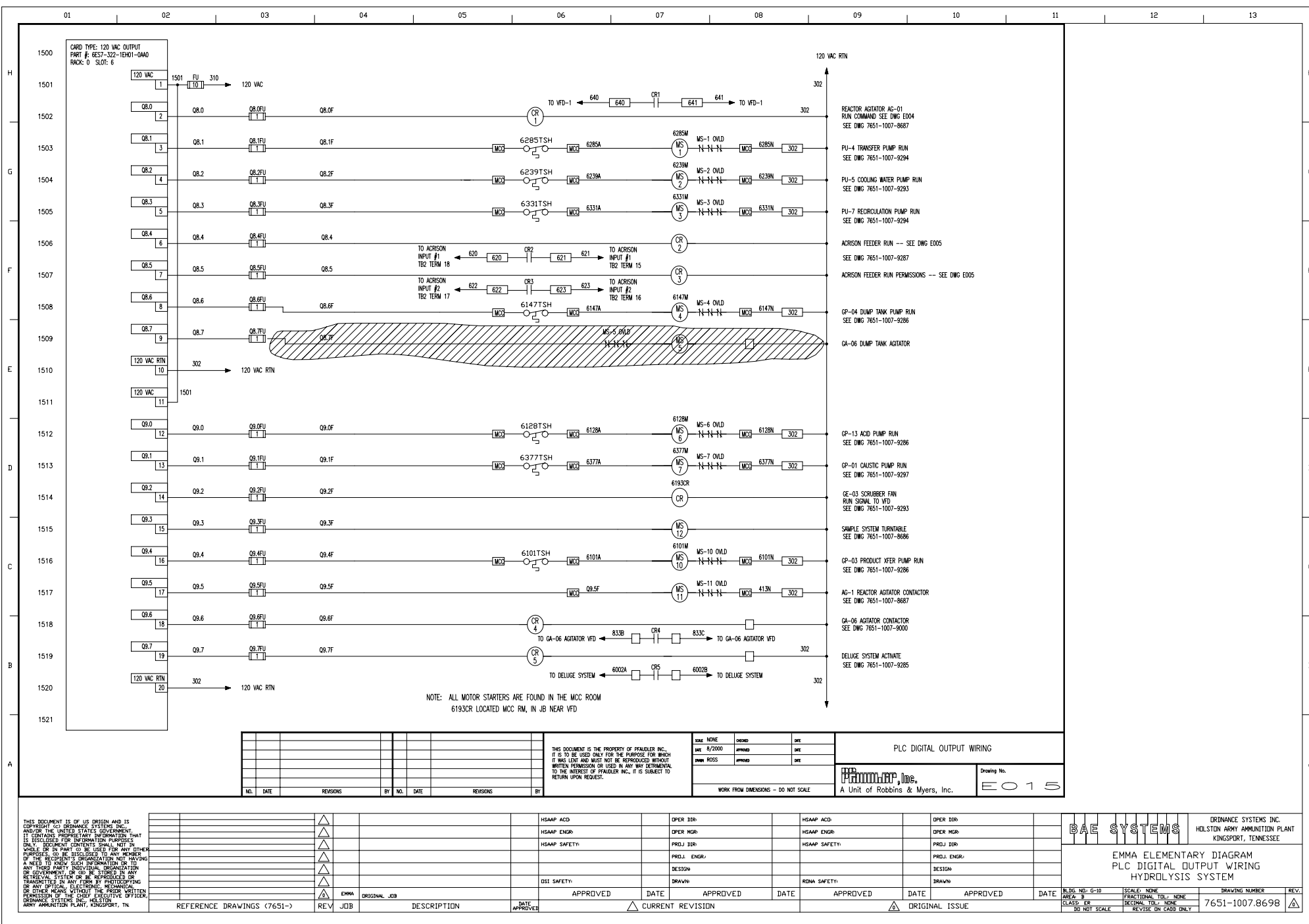




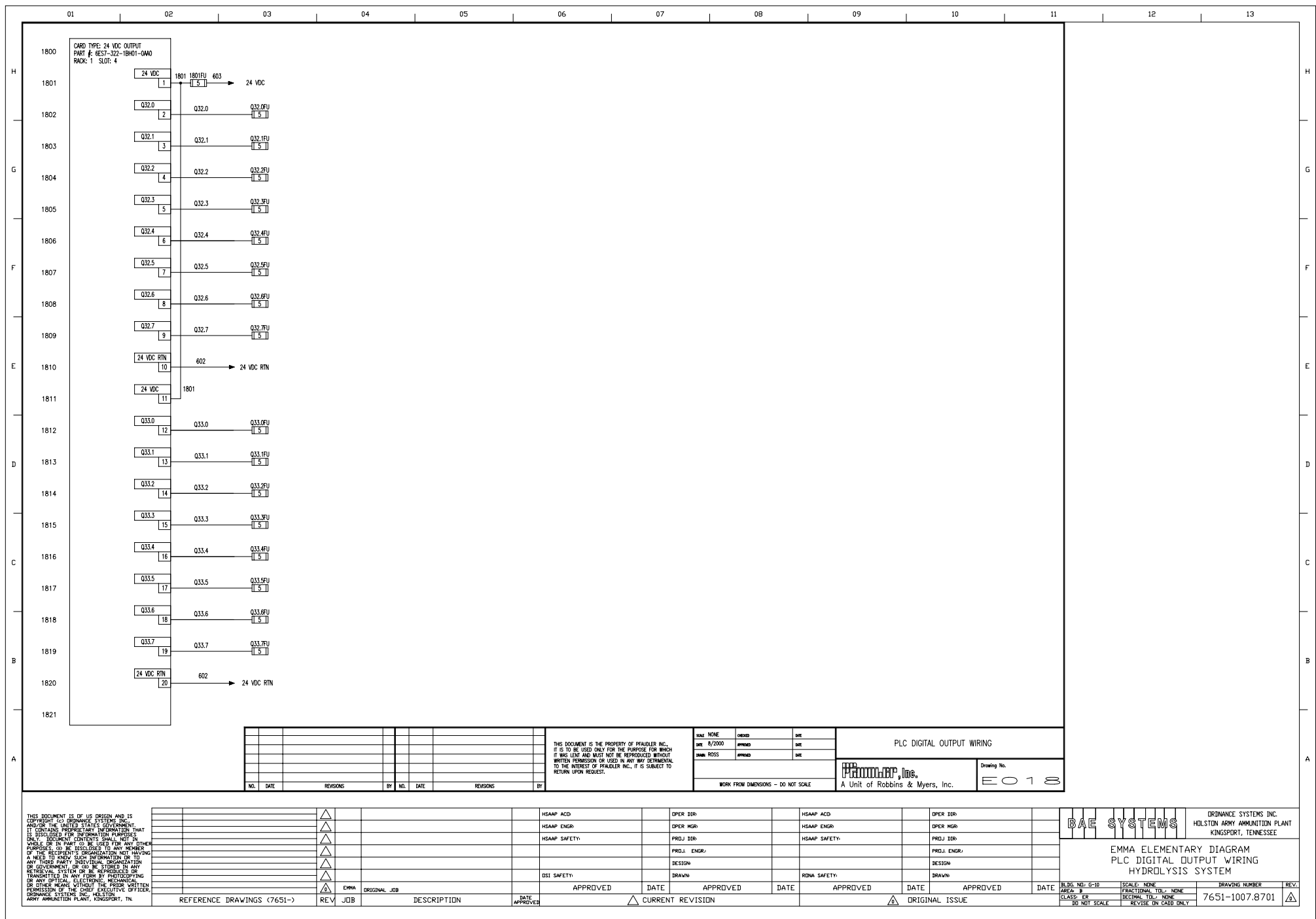


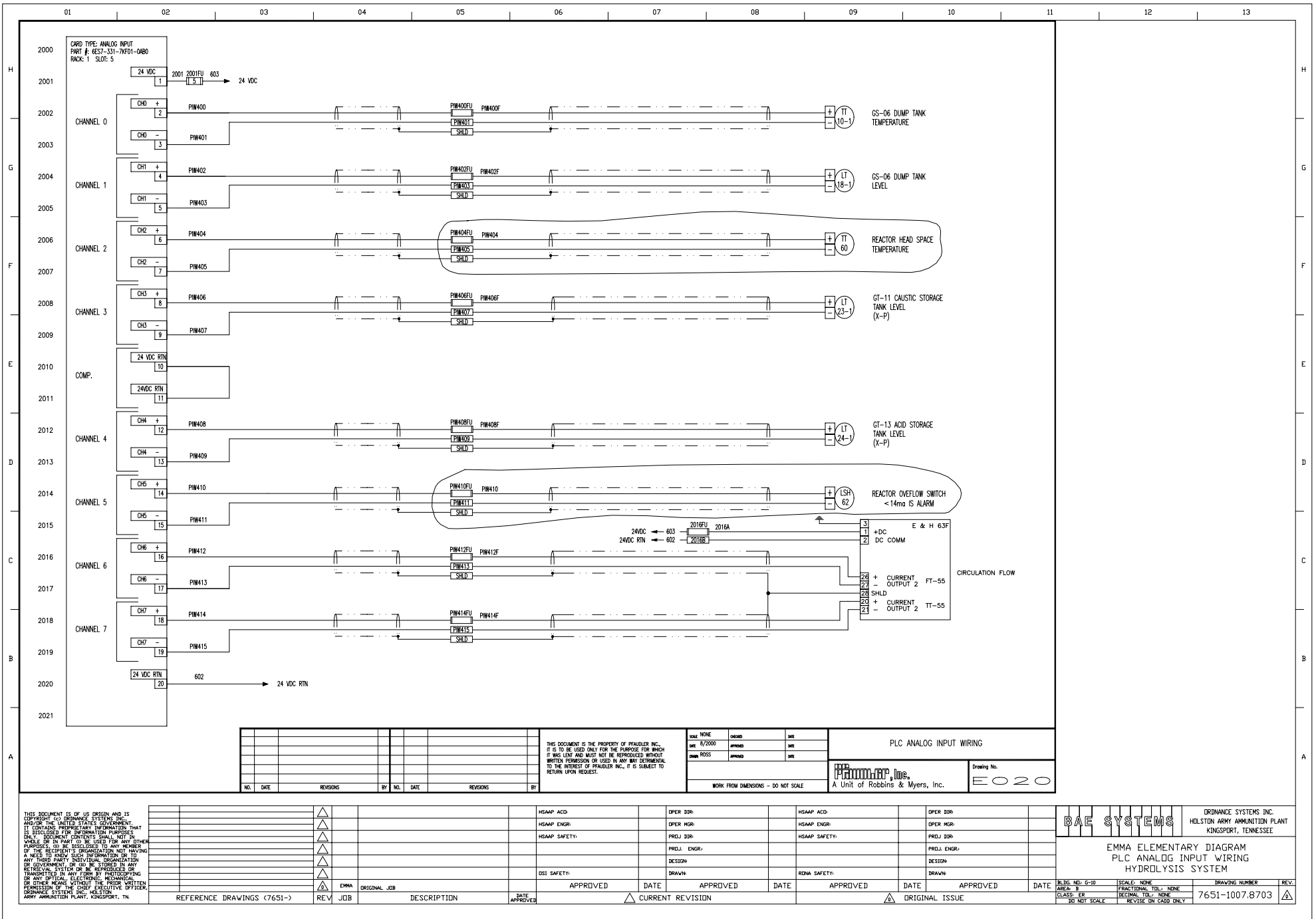


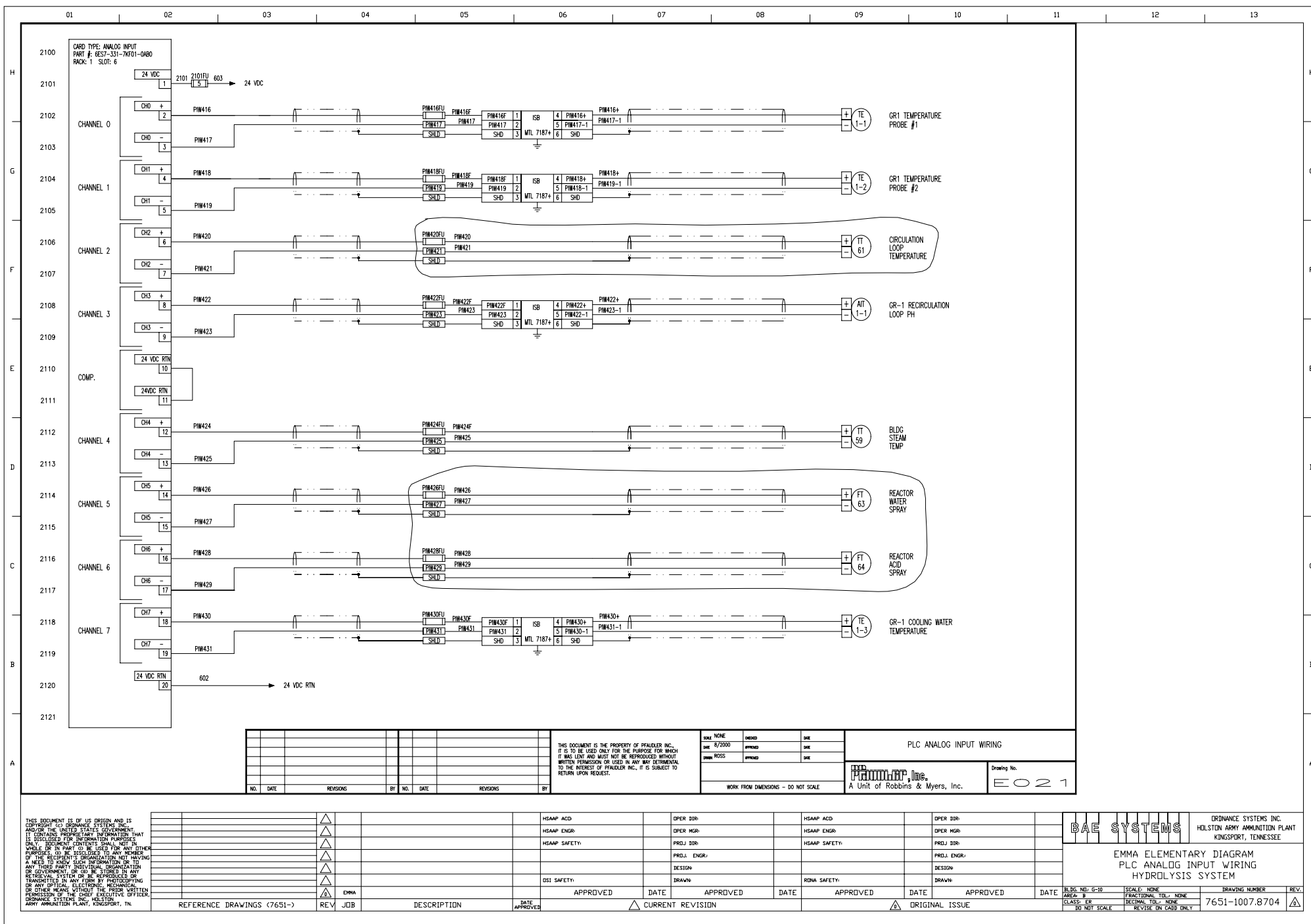






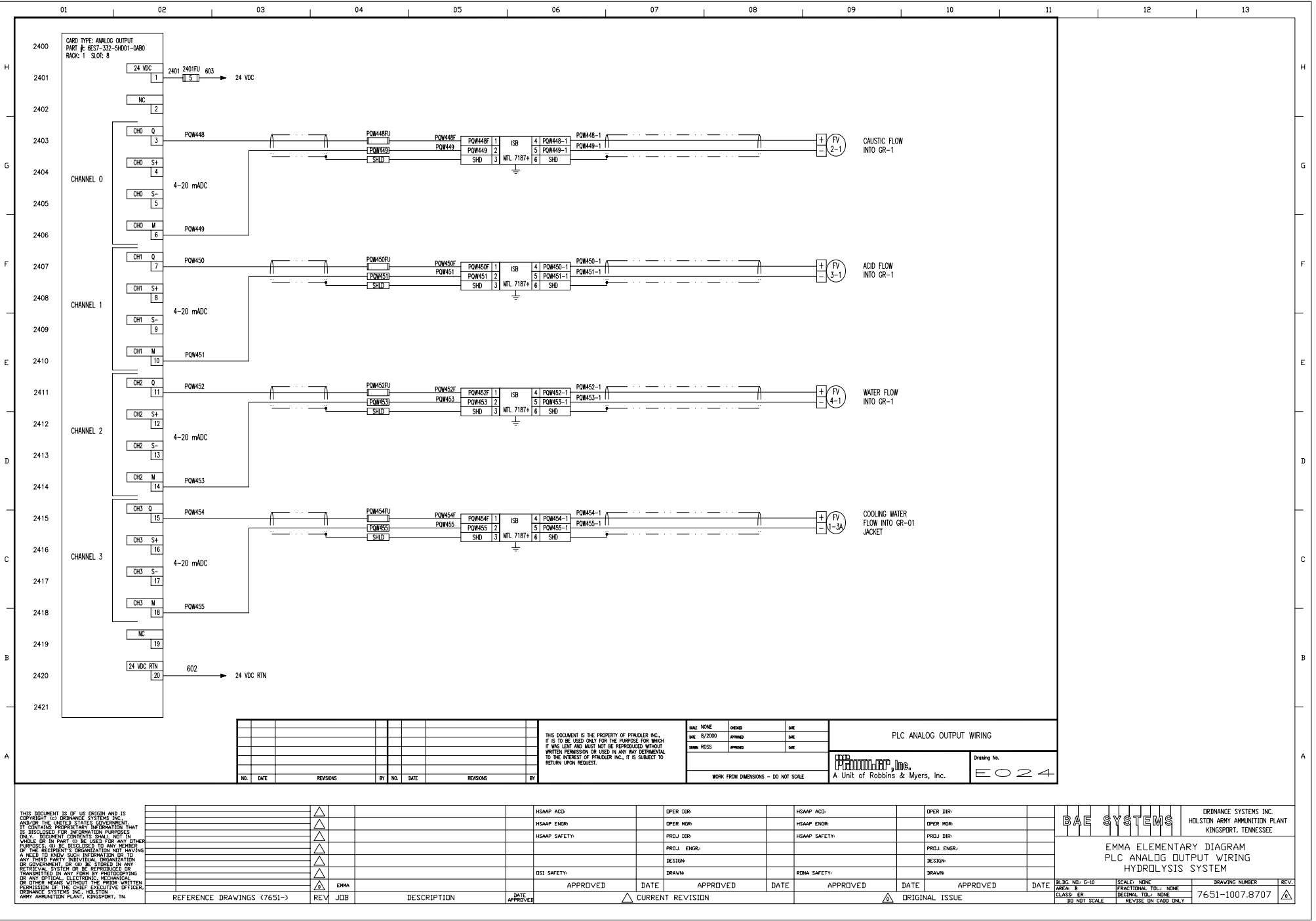






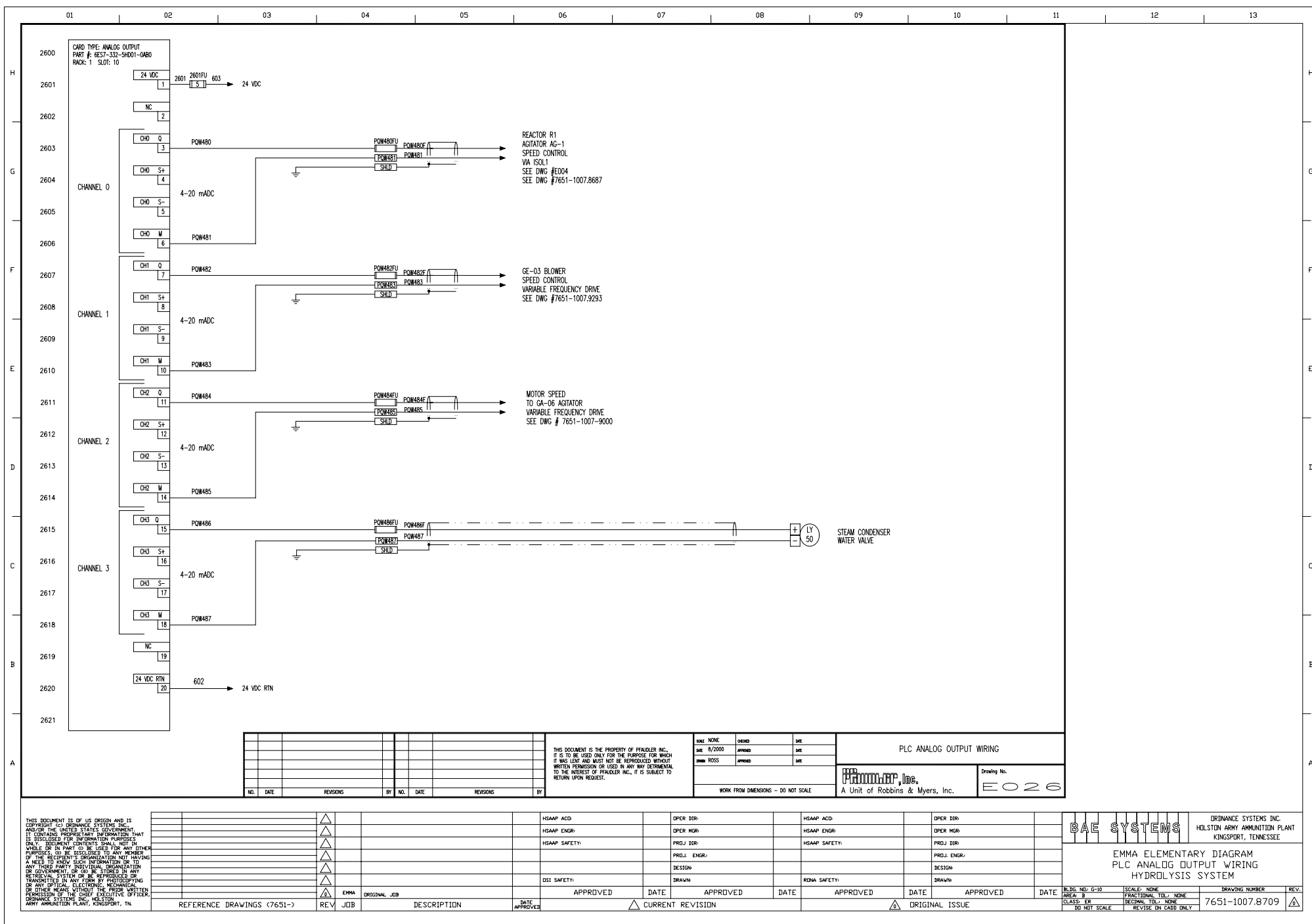




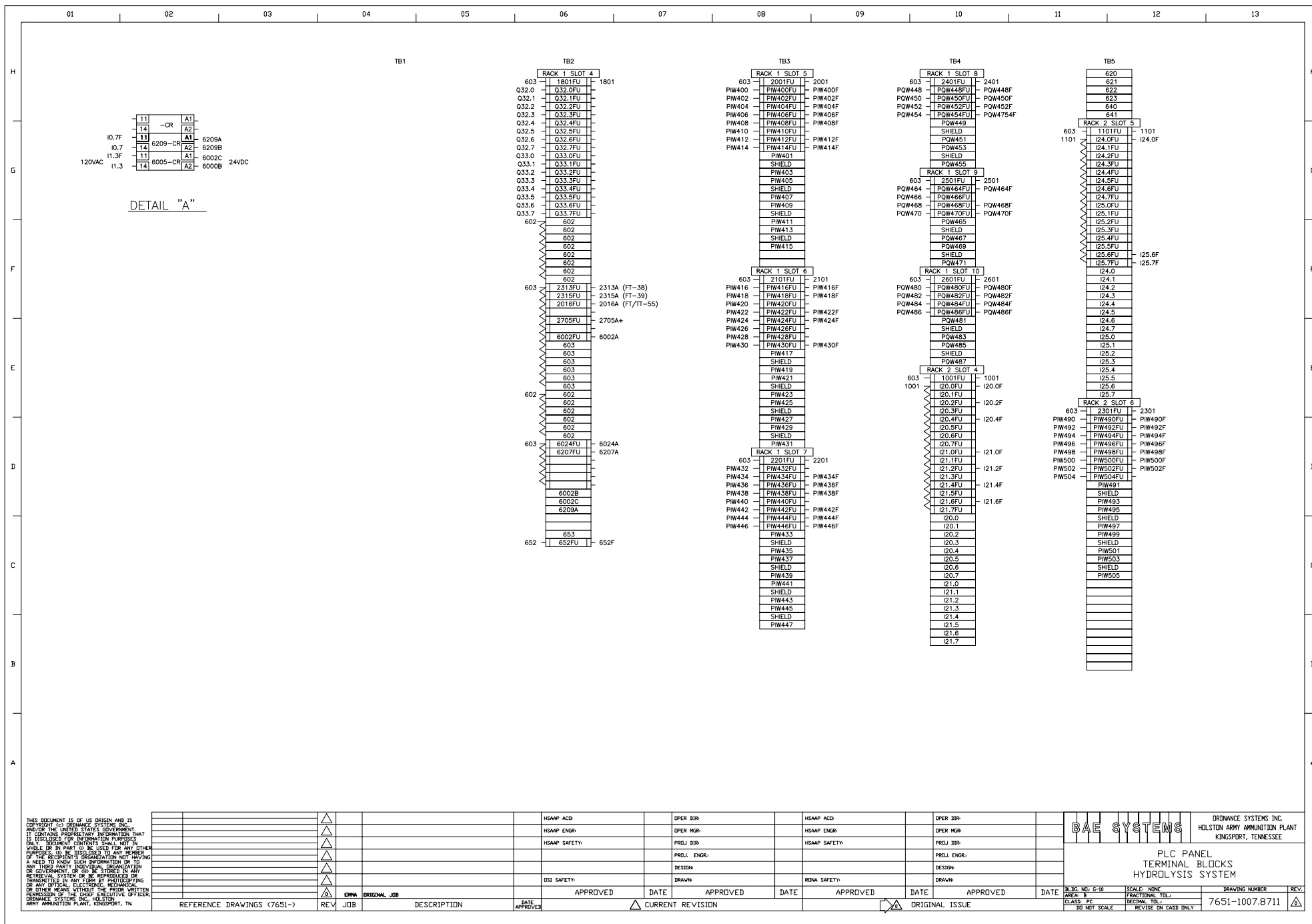


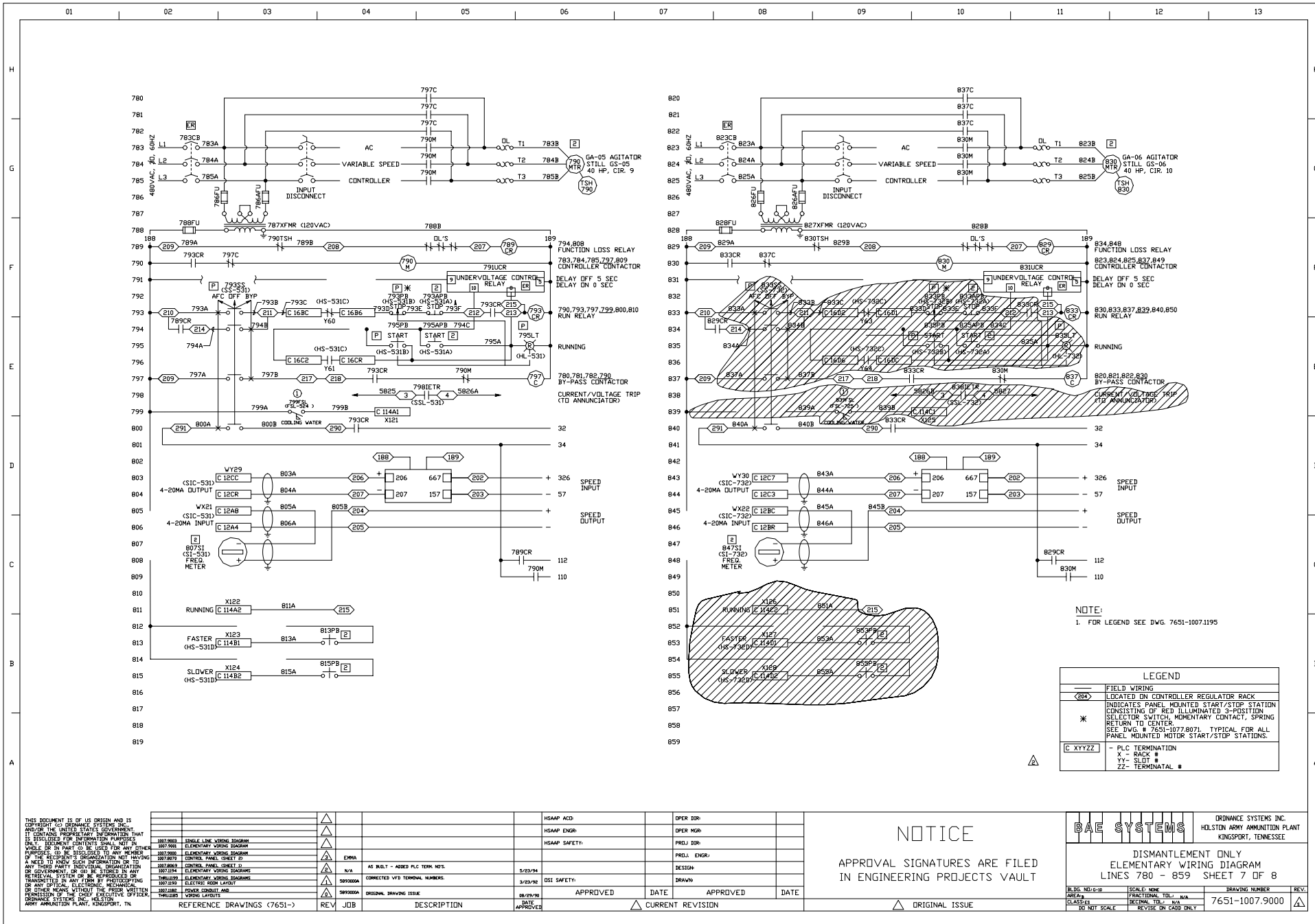


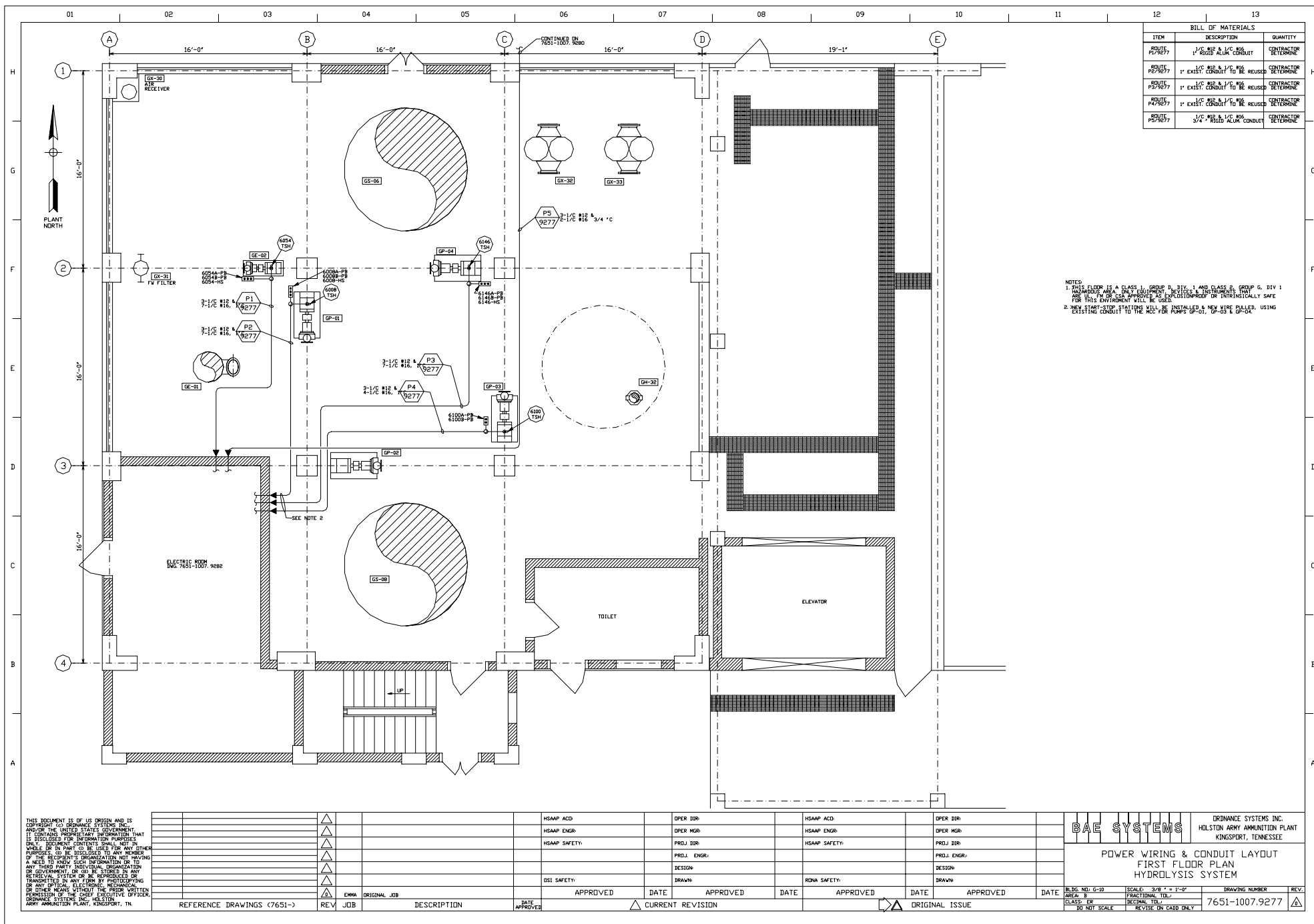


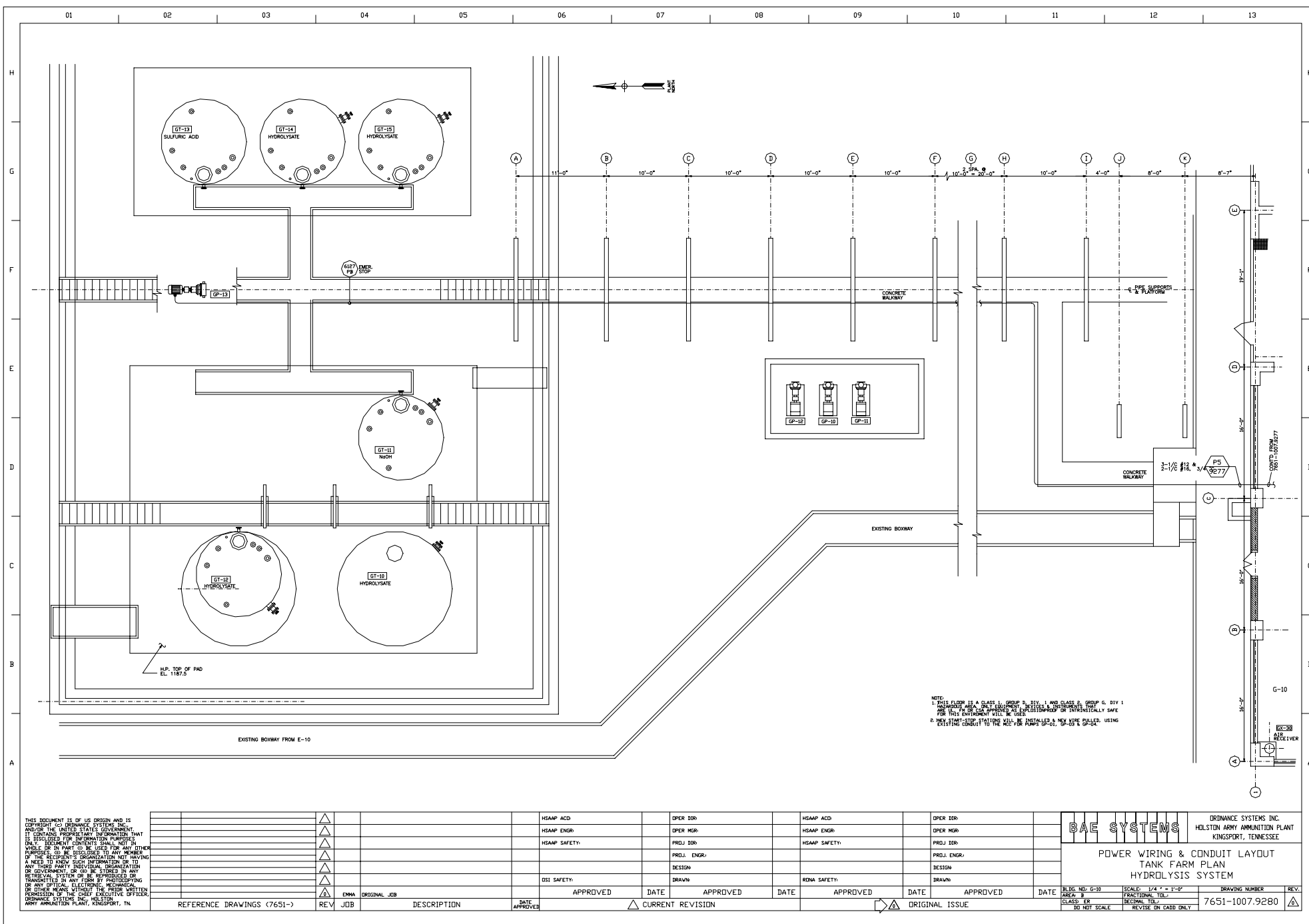






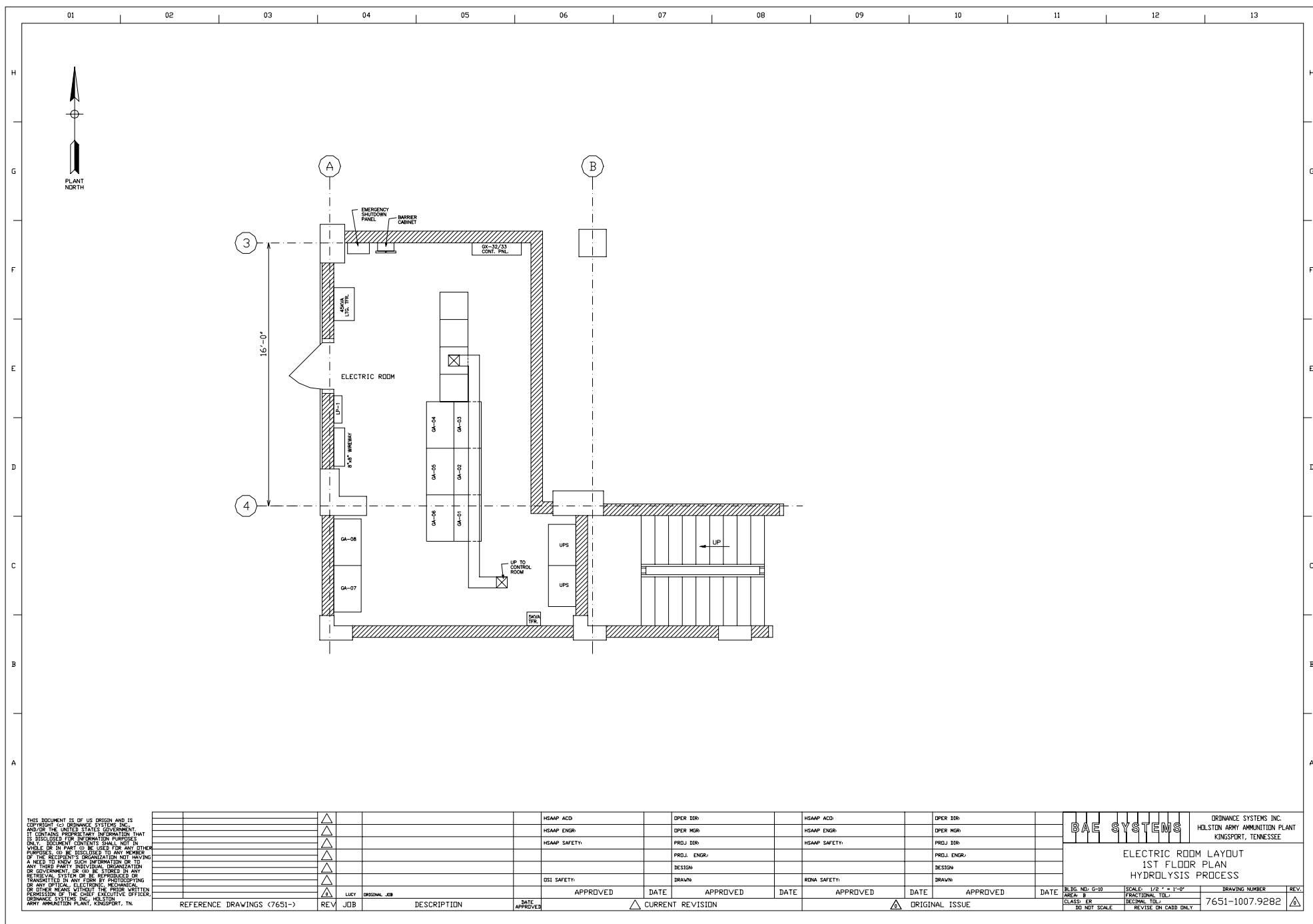


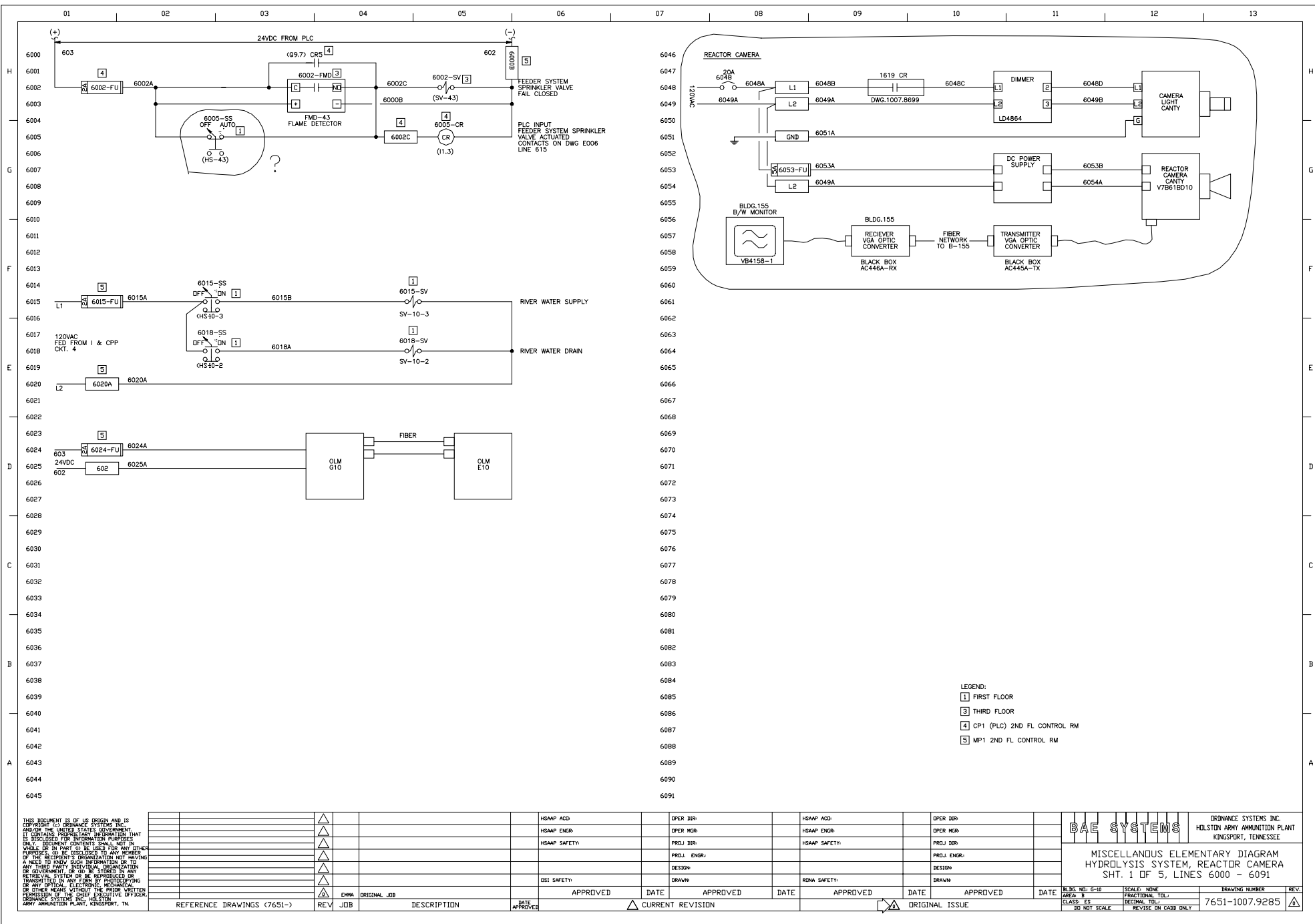


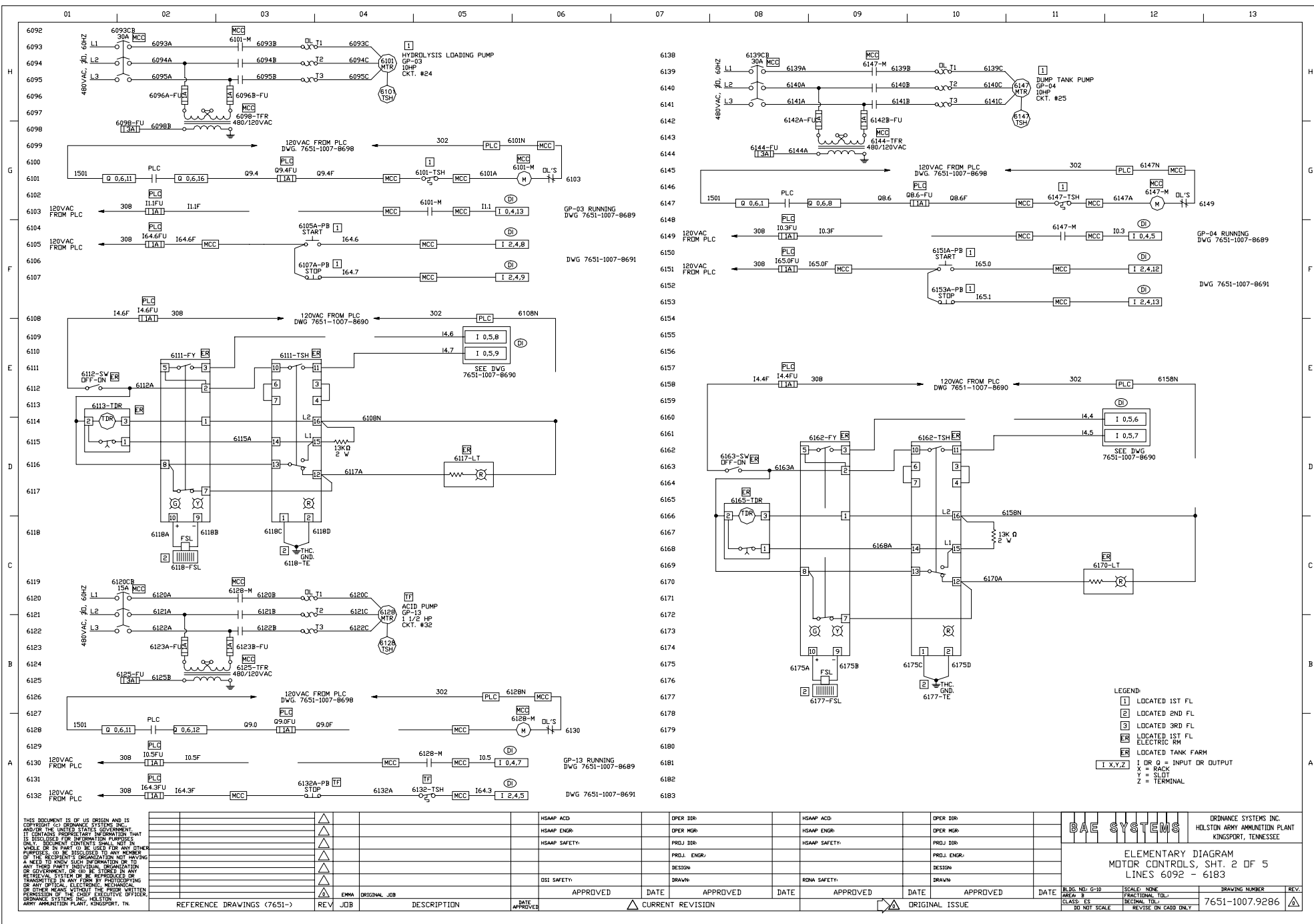


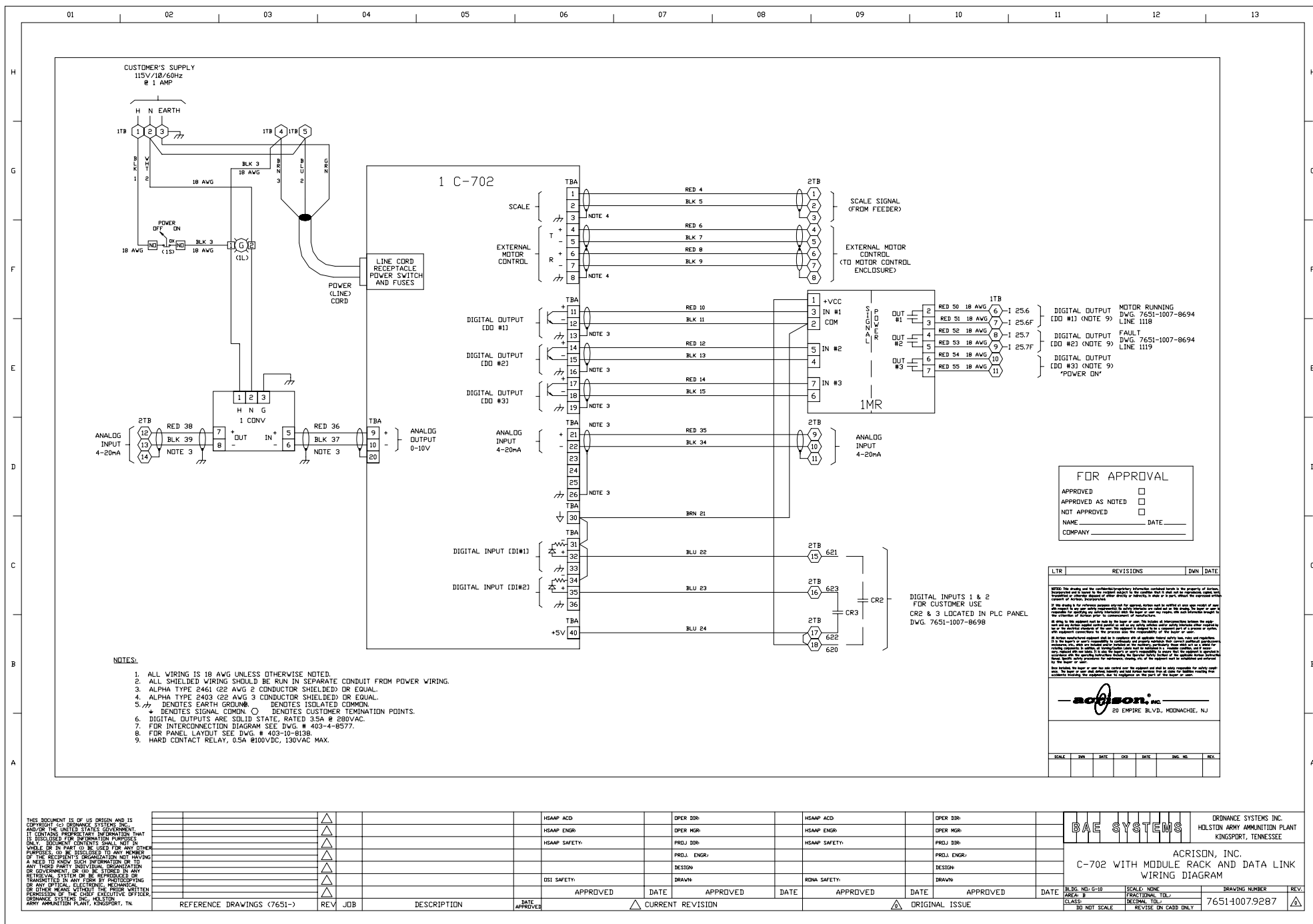




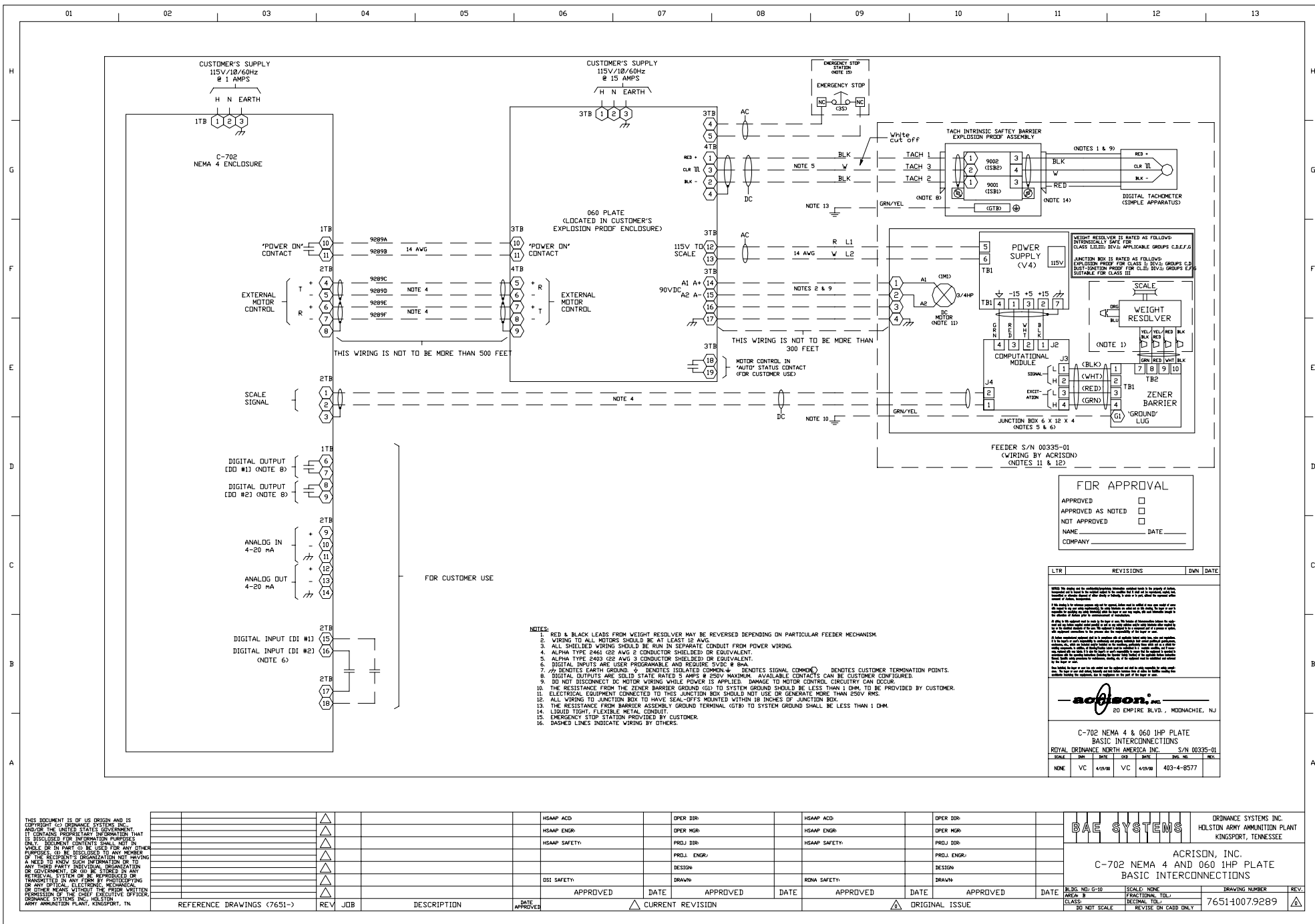


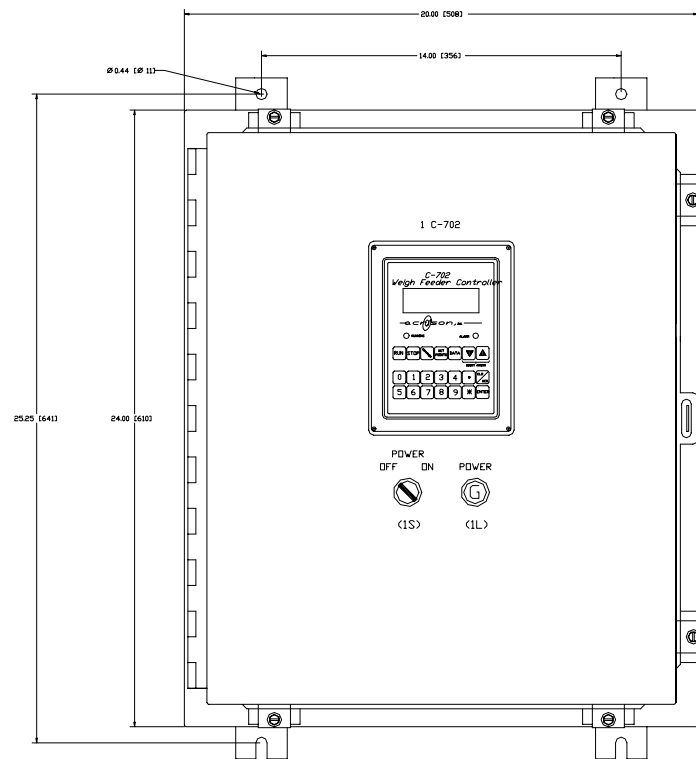




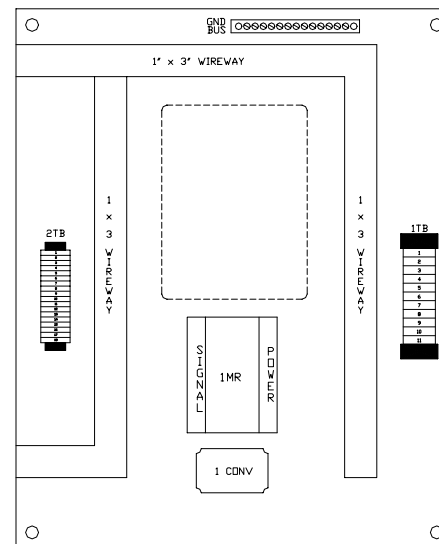








NEMA 4 ENCLOSURE  
24 x 20 x 10  
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PAINT: SEE PARTS LIST



PANEL  
21 x 17  
[533 x 432]

HIGH LEVEL AC/DC WIREWAY

LOW LEVEL DC WIREWAY

FOR APPROVAL

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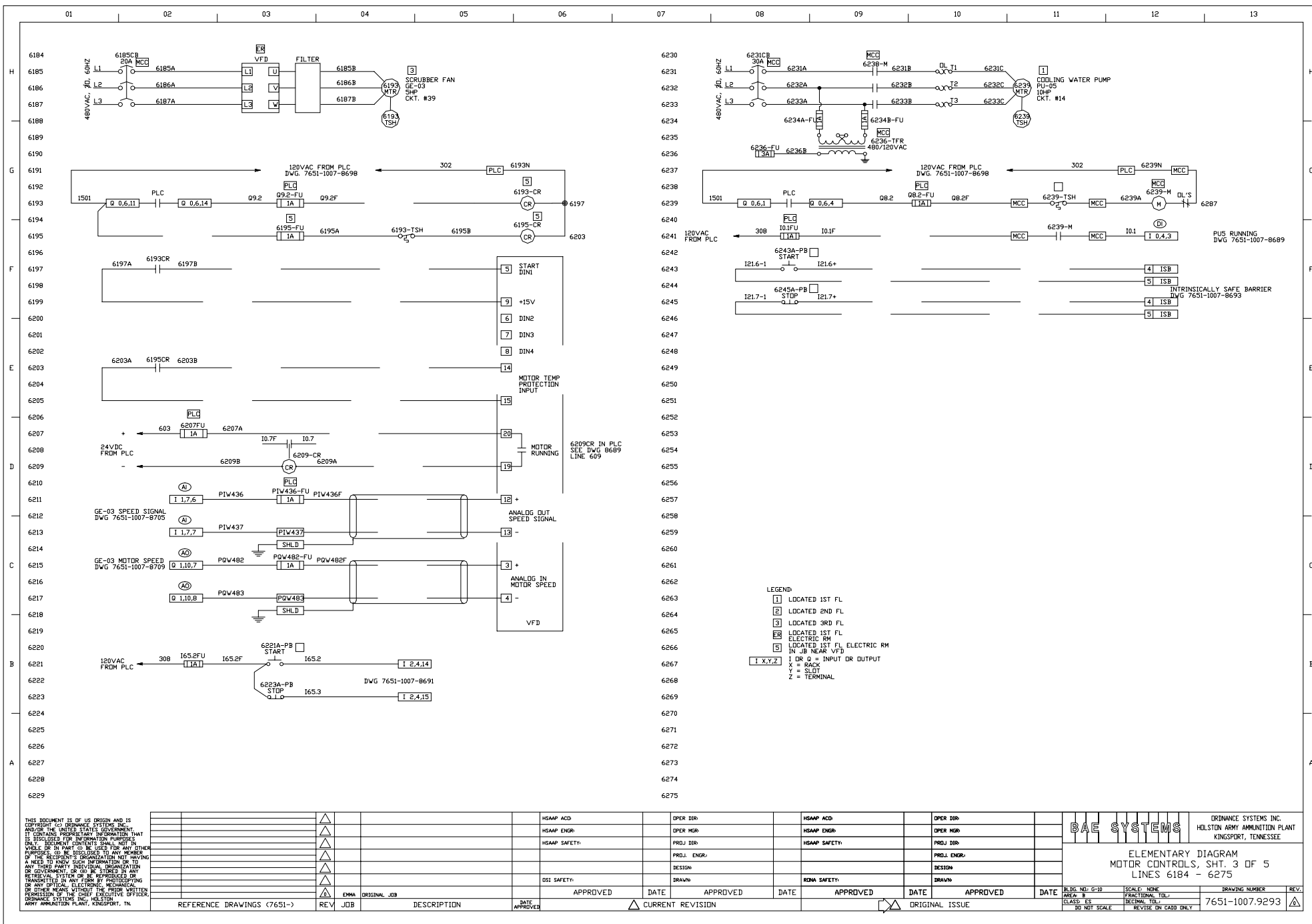
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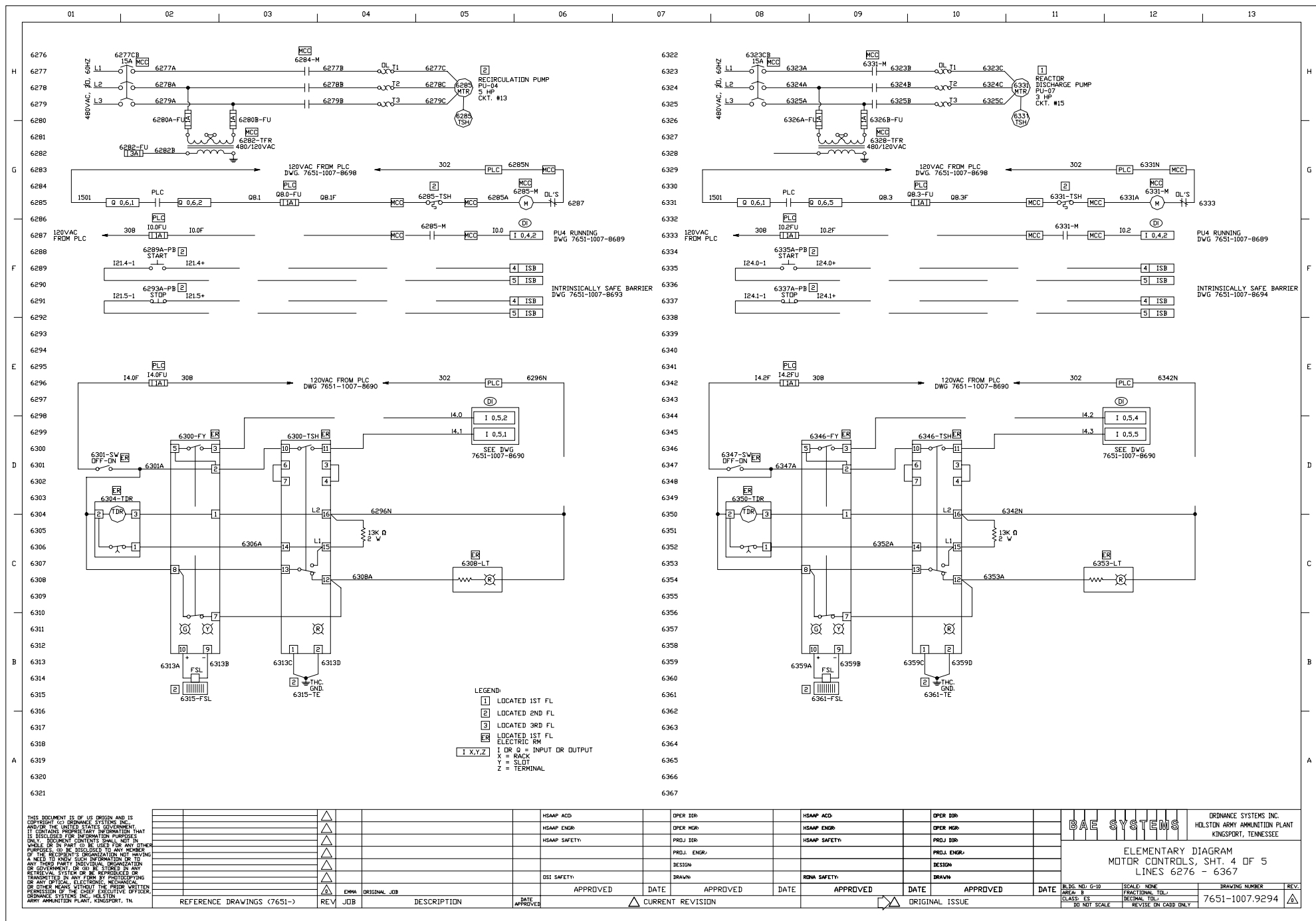
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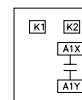
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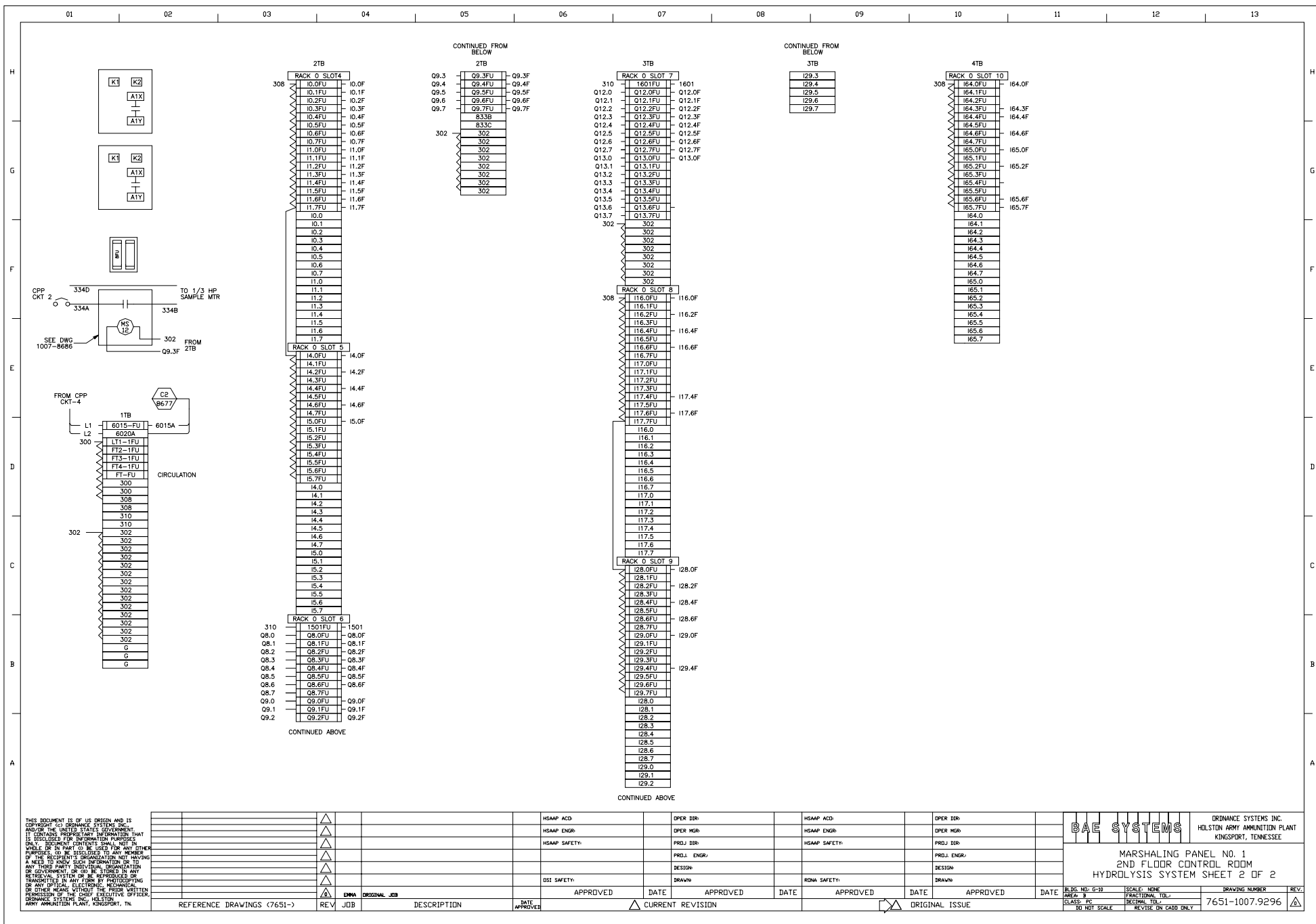


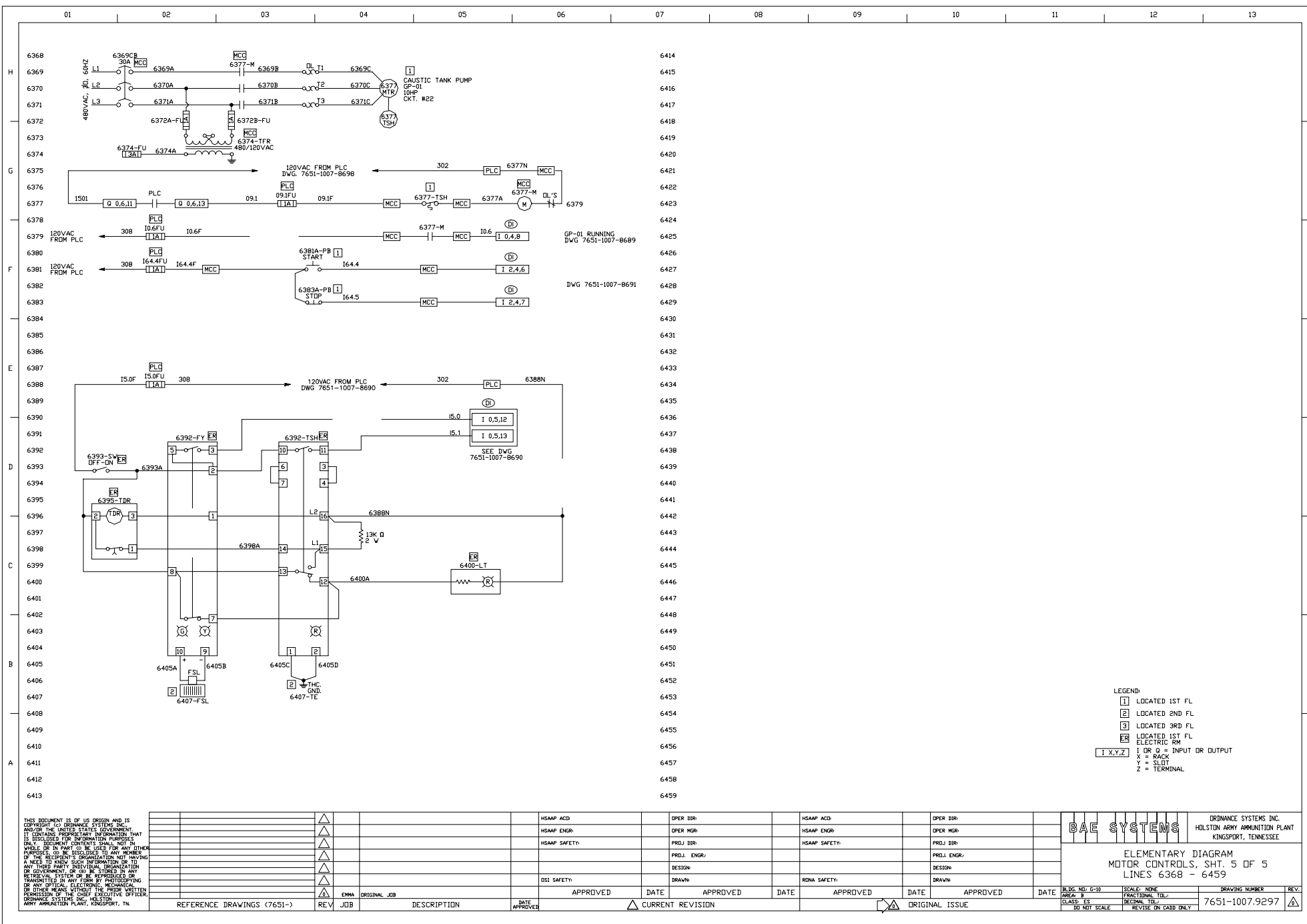




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## **APPENDIX M**

### **PANTEX Report of Composition B and Tetrytol Explosives Hydrolysis Operations**

**Cyclotol and Tetrytol Hydrolysis Operations at  
Pantex Plant**

**Luther Belcher  
Mason & Hanger, Corp.  
A Subsidiary of Day & Zimmerman, Inc.**

**November 8, 2000**

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## **1.0 EXECUTIVE SUMMARY**

### **1.1 Introduction**

In support of the Program Manager for the Assembled Chemical Weapons Assessment Program (PMACWA), seven hundred, twenty pounds of Cyclotol 70/30 and five hundred, forty pounds of tetrytol 75/25 were hydrolyzed with Sodium Hydroxide solutions. The resulting hydrolysate was distributed for use at demonstration test units for energetic hydrolysate destruction efficiency comparison. All of the cyclotol was hydrolyzed with twelve-percent caustic solutions while the tetrytol was hydrolyzed using six- and twelve-percent solutions. The targeted energetic loading rate for most of the work was 16.77%. This loading rate was accomplished with the tetrytol; however, this loading rate was unachievable for the cyclotol due to filtration requirements. All deliverables were successfully accomplished.

### **1.2 Objectives**

The objective for hydrolysate production was to provide demonstration unit feed for incineration alternative evaluation, characterize liquid streams, and gain information on scale-up requirements per PMACWA specifications. The rate and duration of the manufacturing process was based on Pantex Safe operating practices.

### **1.3 Process Summary**

The energetic hydrolysis process utilized 6% and 12% sodium hydroxide solutions for the tetrytol and 12% solutions for all cyclotol, heating this solution to approximately 90 °C, adding the cyclotol or tetrytol, and agitating the solution while maintaining temperature for the time required for energetic destruction. A 200-gallon glass lined reactor was used.

### **1.4 Process Results**

A total of 15 batches of hydrolysate, seven tetrytol and eight cyclotol, were generated and distributed as specified. Energetic concentrations are minimized after one-hour at-temp time for cyclotol and less-than three hours for tetrytol. Liquid and solid samples (filters) were collected and distributed appropriately for analysis. Liquid characterization results will be summarized and provided by A.D. Little representatives. No problems were experienced with tetrytol hydrolysis processing. Foaming in the cyclotol was again a major operational consideration. Destruction removal efficiencies this year were similar to last year's results except in the three batches of cyclotol at 12% where RDX crystals were captured as residue. There are two possible explanations. Approximately two hundred and forty pounds of the cyclotol had two different colored flakes leading to feed composition inconsistencies or the energetic loading rate was too high (too little water was available) and a possible "salting-out" phenomenon increased processing time. Analysis of the different colored flakes was not undertaken due to the possibility that feed inconsistencies are a realistic assumption for processing at a larger scale.

## 1.5 Conclusions and Recommendations

All the objectives for the Pantex portion of this undertaking were accomplished. The hydrolysate was produced and distributed as specified with destruction removal efficiencies similar to last year's effort; except in the case of RDX residue in three cyclotol batches. Filtration was used during Pantex operations and captured unhydrolyzed crystals. This additional operating step should be considered for continued operations. Residue can be cycled into the next batch for continued processing.

Foam was again a controlling factor during operations. Increased agitation rates and energetic addition rate manipulations were used to minimize foaming. Foam, when allowed to blanket the entire liquid surface is a good insulator, and temperature increases become visible. Increased temperature accelerates foam generation, which, in turn accelerates reaction rates. Comp B foaming problems were assumed to be associated with the wax; however, Cyclotol, with Calcium Silicate, demonstrated similar foaming tendencies. Further operations should be conducted with foam consideration/control built into operations.

Solid minimization in cyclotol processing must be ensured, through longer at-temp times, liquid/caustic level maintenance, and/or hydrolysate filtration. Neutralization and filtration were Pantex-specific operating procedures.

Cyclotol destruction is maximized in one-hour; however, all Pantex batches were held for two hours at-temp during Pantex processing. HPLC results were used to verify the minimization of energetic concentrations.

Scale-up parameters for safe operations include, but are not limited to, caustic concentration, energetic loading rates, temperature, feed rate, physical configuration of explosive, and solution agitation and baffling mechanism(s).

Tetrytol handling and reactor addition must be carefully monitored to ensure that impact sensitivity concerns are addressed, as the drop-height is similar to HMX. Tetrytol is a clean hydrolyzing material with no foaming, exothermic, or solids-forming tendencies. Due to material form, dissolution should be ensured through addition rate manipulation to avoid a two-phase potential (material sinks to the bottom before melting and is not dispersed). Sufficient agitation, operating temperatures, and addition rate considerations will help alleviate this potential.

Tetrytol hydrolysate will foam excessively if the pH is five or less. Care must be used if neutralization is used. Energetics destruction was maximized in less-than three hours. The first sample was taken at three hours due to required safe liquid-level operating requirements (water level higher than twelve inches above blades). The excess water was evaporated to meet PMACWA's energetic loading requirements.

## **2.0 INTRODUCTION**

The Pantex Plant in Amarillo, Texas manufactured Cyclotol and Tetrytol hydrolysate. This work was conducted in support of the Program Manager for Assembled Chemical Weapon Assessment (PMACWA) Demonstration Test Program.

The PMACWA Demonstration Test Program was established as a result of Public Law 104-208, under which the Under Secretary of Defense for Acquisition and Technology appointed PMACWA with the mission to demonstrate alternate technologies to “baseline” incineration for the disposal of assembled chemical weapons. The purpose of this Test Program is to select and demonstrate approaches, other than the “baseline” incineration, that provide complete demilitarization and disposal of the assembled chemical weapons stored in the United States’ chemical weapons stockpile.

PMACWA was responsible for the production of energetic hydrolysate solutions as feed to technologies designed to treat such solutions. Energetic hydrolysate solutions generated for the PMACWA program at Pantex were sodium hydroxide solutions 6- and 12 percent for tetrytol and 12 percent for cyclotol.

This report describes the activities associated with the production of Cyclotol and Tetrytol hydrolysate solutions at the Pantex Plant.

## **3.0 OBJECTIVES**

The objectives of the energetic hydrolysis production consisted of the following:

- Manufacture greater-than 5150 pounds hydrolysate from 720 pounds cyclotol (Stock material).
- Manufacture greater-than 3650 pounds hydrolysate from 540 pounds tetrytol (Stock material).
- Hydrolysate delivery to specified locations.
- Sampling and distribution of materials as requested.
- Rate and Duration of manufacturing process based on Pantex safe practices.

## **4.0 OVERVIEW OF HYDROLYSATE PRODUCTION SYSTEM**

Initial Safety requirements included MSDS recommended PPE in addition to task exhaust for vapor and dust control. Considerations for the handling of solutions with elevated pH and handling solids with drop height considerations (padding hard surfaces) were built into

processing operations. Foam and temperature excursion considerations were also incorporated into daily operations.

The hydrolysis reaction was performed in a 200-gallon glass-lined Pflaudler™ chemical reactor (Figure 1). The reactor vessel is equipped with an agitator, heating/cooling jacket, temperature monitoring equipment, and view ports. The system consists of the following major pieces of equipment:

- Hydrolysis Reaction Vessel (Pflaudler™)
- Deionized Water Supply
- Temperature Control System
- Off-gas handling Systems
- Hydrolysate Filter System (cartridge filter)
- Hydrolysate Receiving Vessels (55 or 30 gallon drums)

The energetics used in the Pantex hydrolysis process was Cyclotol and Tetrytol. The Cyclotol was a reddish-white platelet approximately 1/8" thick with varying shapes and sizes from 1/4" to 3/4". It contains a mixture of approximately 70% RDX, 30% TNT and no more than 0.5% calcium silicate. The RDX also contains 5% to 20% of HMX as an impurity, which also has energetic properties. The tetrytol used was obtained from demolition blocks that were broken up into pieces weighing up to 1/2 pound. The material was yellow and consists of a mix of approximately 75% Tetryl and 25% TNT. Hydrolysate solutions to be produced were:

6% solutions totaling: 2211 lbs. hydrolysate from 147-lbs. tetrytol

12% solutions totaling: 4180 lbs. hydrolysate from 720-lbs. cyclotol  
2240 lbs. hydrolysate from 393 lbs. tetrytol

#### **4.1 Description of the System**

The process used standard chemical operating facility equipment. This process was conducted during normal working hours. Each batch required approximately 4 days to complete and required the reactor to be cooled at the end of each working day. The process steps were as follows:

- Water and sodium hydroxide (NaOH) were added to the vessel to make either a 1.5 or 3.0 molar NaOH solutions.
- The solution was slowly heated to a maximum of 90 °C. During processing, the vessel agitation is used to stir the vessel contents, maintain homogeneity in the solution, prevent hot spots, and control foaming. Controlling the rate at which steam was added to the vessel

jacket regulated the temperature. At no point during the operation was the solution temperature to go above 95 °C.

- Cyclotol or Tetrytol were added to the vessel at a rate commensurate with the experience and knowledge gathered during similar operations conducted last year. Four hours was the minimum addition timeframe.
- Solution temperature was maintained at operating temperature for a sufficient time period to destroy the energetics and then cooled
- The hydrolysate was neutralized and filtered before being packaged for shipment. The 12% hydrolysate was neutralized with phosphoric acid and the 6% hydrolysate was neutralized with sulfuric acid. The filtered hydrolysate was then transferred into 55 or 30-gallon drums for shipment to the demonstration sites.

## **5.0 HYDROLYSIS OPERATIONS**

Handling precautions were the same as last year's operations. Specified PPE was used in accordance to MSDS guidance. Local ventilation was used whenever explosives were handled and whenever vapors were emitted. The tetrytol was considered a consolidated explosive and required padding to be installed over all hard surfaces wherever the pieces were to be handled. To avoid agitator impingement, a minimum of 12-inches of water was maintained between the top of the blades and the liquid surface. To alleviate any potential exotherm, a four-hour minimum timeframe for energetic addition per batch was utilized. Operating temperatures were maintained less than 90 C.

Operations are normally conducted using 1.5- or 3-molar concentrations with molar equivalencies of 4.5 moles of Sodium Hydroxide per mole of energetic. For the cyclotol, this is approximately a 7.5% loading rate at 1.5 molar and approximately 15% loading rate at 3 molar.

Five batches of cyclotol were run using 81 gallons of water, 81 pounds of NaOH, and 97-98 pounds of cyclotol. From last year's work, two conditions were to be avoided: uncontrolled exothermic conditions and uncontrolled foaming. PMACWA specified that no chemicals were to be added to help with foam mitigation. The total batch energetic weight was divided by 48 (4 hours at 5-minute intervals) to get the value for a single addition. This year, the energetic was added over a 6.5-hour timeframe. Energetic addition was accomplished by making an addition, waiting five minutes, adding another, waiting ten, adding one, wait five, add another, wait ten until ten additions were made. At that point, a thirty-minute time span with no additions was allowed to elapse. Another mechanism utilized for foam abatement was agitation. Using addition rate, the thermowells as a baffle, and severe vortexing action, the foam was not a

problem. The reactor utilized had an approximate 40" diameter with increased surface area due to liquid vortexing. On average, the addition rate was 13-pounds/hour/15.9ft<sup>2</sup> surface area.

All of the tetrytol hydrolysate was manufactured with no operational difficulties. Extra water volumes were used to ensure the twelve-inch liquid height above the agitator blades. At the end of energetic addition, water was allowed to evaporate until the desired loading rate was obtained. At that point, the hydrolysate was neutralized, filtered, and drummed for shipment.

## **6.0 RESULTS**

One area that differed in this year's operations was the specified hold time for the hydrolysate. This hold time began after the last energetic addition. Hold time for FY99 work was specified at 8 hours. This year the hold time was left to be determined. During the first batch of Cyclotol, at the end of the energetic additions, samples were taken at one-hour intervals, neutralized, and analyzed. It was demonstrated that at the end of one hour, the energetic concentrations were minimized. Results demonstrated that hold-time after the one-hour did not improve destruction rates. Subsequent batches were held for two hours at-temp before neutralization. HPLC analysis of each batch verified destruction levels.

Foaming during cyclotol processing needs to be avoided. Process observation, through temperature monitoring and visual inspections of the solution during additions, was utilized to avoid foaming conditions. Temperature considerations become apparent whenever the foam covers the entire liquid surface (foam is a good insulator). Reaction rates are enhanced at higher temperatures. Ninety degrees Celsius, plus/minus three degrees, was the specified operating temperature for all Pantex operations. In reality, all operations were kept as close to, but below, ninety degrees. There seemed to be two types of foam. There was white-colored foam generated from normal processing. It appeared approximately twenty-minutes after an addition. This foam was extremely water-soluble and would last approximately forty-five minutes. The other type of foam, red-colored foam, appeared after many additions. It seemed that this foam was released from a gas-saturated solution. This foam was less water-soluble and extremely persistent, lasting approximately one hour. Boiling the cyclotol hydrolysate also leads to excessive foam generation.

Solids generation in the cyclotol hydrolysate became apparent during the first batch. In trying to reach the required 16.77% energetic loading rate, the salt precipitate was sufficient to impede filtration. All solids collected during this batch were completely water-soluble. Three of the last four batches had noticeable solids that were not dissipated with Deionized water. The energetic loading for these three batches were 28%,

**Figure 1: 200-Gallon Reactor**



19% and 18%. After the solids generation in the 28% and 19% batches, a subsequent batch was held at an approximate 14% energetic loading and zero solids were generated. If sufficient water is available during the hydrolytic destruction, energetic constituent destruction efficiency is maximized and crystal formation is minimized. If the water level becomes insufficient (energetic concentration becomes too high), some of the larger crystals may not be processed. It is believed that the Asalting-out@ phenomenon becomes prevalent at hydrolysis operations with insufficient water. If the salt concentration in the solution becomes too high, salts impede hydrolytic destruction. This phenomenon has been observed for other energetic materials.

Two hundred, forty pounds of the cyclotol had two different colored flakes in the box, some reddish-white and some white. The different flakes were not analyzed because all of the feedstock was from the same Holston lot. If the crystals were from the different colored flakes, then realistically, a larger-scale operation could encounter similar materials. The formation of crystals in the hydrolysate was noted and reported for that reason. These crystals were rinsed off of the filters into the next batch for continued exposure to the caustic solution. Crystal assay results demonstrated almost pure RDX. Continued caustic exposure was ineffective in destroying these crystals once they were formed.

Tetrytol is a different story. Due to the required 12@ liquid buffer, processing was accomplished at well below the specified 16.77% energetic loading. Upon energetic addition completion, the required loading was accomplished by evaporating the water. Due to the time required for the evaporation, the first sample taken of the tetrytol hydrolysate was at three hours and the next at five hours. The three-hour sample demonstrated that energetic concentrations were minimized. This method was used for the balance of this year=s operations.

Viscosity measurements were taken of both hydrolysates. The results were 1.4186 cp (mPa-s) for the cyclotol hydrolysate at 16.77% loading and 30C. Viscosity for the tetrytol hydrolysate was 1.411cp for the tetrytol hydrolysate at 12.5% loading.

Energetic concentration levels in all cyclotol hydrolysate manufactured were below 1.6 ppm and were below 0.5 ppm as determined through HPLC analysis. This compares directly with the destruction removal efficiencies from the previous year's work.

## **7.0 SUMMARY**

Hydrolysis operations for Cyclotol and tetrytol were accomplished safely with results similar to last year's. Foam considerations during cyclotol operations must be incorporated into operating system design, either through minimization or control upon generation. Operating temperature control is directly influenced by foam generation. Filtration (5-micron) and neutralization were Pantex specific operating steps; however, filtration should be considered for solids removal. Energetic loading rates above approximately 14% for cyclotol lead to solids formation with salt formation occurring up to approximately 17% and energetic solids residue at concentrations



above the 17%. Tetrytol hydrolysis operations experienced no operational difficulties. Safe handling considerations are required due to material form and drop height considerations. Tetrytol hydrolysate will foam rapidly (off-gas) at pH levels 5 and below.

## **APPENDIX N**

**Test Plan Requirements (TPR), Characterization of an Energetic Hydrolysis Reactor System at  
Holston Army Ammunition Plant/BAE Systems**

Date: June 18, 2001

# TEST PLAN REQUIREMENTS

## CHARACTERIZATION OF AN ENERGETIC HYDROLYSIS REACTOR SYSTEM AT HOLSTON ARMY AMMUNITION PLANT/BAE SYSTEMS

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## Test Plan Requirements (TPR) For Characterization of an Energetic Hydrolysis Reactor System

### OBJECTIVE

The objective of the TPR is to clearly determine and define the optimum operating parameters for the energetics hydrolysis process to support the design and installation of the full-scale hydrolysis process at the Pueblo Chemical Agent Disposal Facility and the Lexington Bluegrass Chemical Agent Disposal Facility for the destruction of the energetics contained in the following munitions:

Location	Munition Item	Propellant	Burster	Igniter/Charge
<b>Pueblo Chemical Agent Disposal Facility</b>	4.2-inch Cartridge, Chem Agent HT, M2	5 Incr. Bundle: 0.32 Lb M8 Prop ½ Incr.: 0.008 Lb M8 Prop 1 Incr.: 0.08 Lb M8 Prop	Tetryl – 1003 grains (64.9944 grams)	Prop Chg: 120 grains (7.776 grams) M9 Prop
	4.2-inch Cartridge, Chem Agent HD, M2A1	5 Incr. Bundle: 0.32 Lb M8 Prop ½ Incr.: 0.008 Lb M8 Prop 1 Incr.: 0.08 Lb M8 Prop	Tetryl – 1003 grains (64.9944 grams)	Prop Chg: 120 grains (7.776 grams) M9 Prop
	105-mm Cartridge, Chem Agent HD, M60	Zone 1: 8.6 OZ M1 Prop Zone 2: 1.4 OZ M1 Prop Zone 3: 2.5 OZ M1 Prop Zone 4: 3.8 OZ M1 Prop Zone 5: 5.8 OZ M1 Prop Zone 6: 8.8 OZ M1 Prop Zone 7: 14.3 OZ M1 Prop	Tetrytol – 1800 grains (116.64 grams)	Black Powder - 300 grains (19.44 grams)
	155-mm Projectile, Chem Agent HD, M104		Tetrytol – 2900 grains (187.92 grams)	
	155-mm Projectile, Chem Agent HD, M110		Tetrytol – 2900 grains (187.92 grams)	
<b>Lexington Bluegrass Chemical Agent Disposal Facility</b>	155-mm Projectile, Chem Agent HD, M110		Tetrytol – 2900 grains (187.92 grams)	
	8-inch Projectile, Chem Agent GB, M426		Comp B4 – 7.0 Lb.	
	115-mm Rocket, Chem Agent GB, M55	M28 Grain– 19.1 Lb	Comp B w/Ca Silicate – 3.14 Lb	
	115-mm Rocket Warhead, Chem Agent GB, M56	M28 Grain– 19.1 Lb		Comp B w/Ca Silicate – 3.14 Lb
	155-mm Projectile, Chem Agent VX, M121/A1		Comp B4 – 2.45 Lb	0.30 Lb Sup Chg – 98.5% TNT & 1.5% Ba Stearate
	115-mm Rocket, Chem Agent VX, M55	M28 Grain– 19.1 Lb	Comp B w/Ca Silicate – 3.14 Lb	
	115-mm Rocket Warhead, Chem Agent VX, M56	M28 Grain– 19.1 Lb	Comp B w/Ca Silicate – 3.14 Lb	

**Figure 1. Summary of Energetics Contained in Stockpile Munitions**

This Plan will also address and integrate the results of bench-scale testing to be conducted by Los Alamos National Laboratory (LANL) and the Naval Surface Warfare Center (NSWC) to respond to the National Research Council (NRC) concerns regarding:

- Solubility of energetics in specific alkaline solutions,

- Simultaneous processing of different types of energetics, and
- Particle size reduction of energetics that must be achieved for proper operation.

LANL will:

- Fully characterize the hydrolysis of energetic material on a bench scale by fully analyzing the hydrolysate and the off-gas generated from the various energetic material processing,
- Determine the optimum caustic soda concentration, which will insure maximum destruction efficiency at a minimum reactor residence time by hydrolyzing at various concentrations (12, 20, 25, and 30 wt.%), and
- Investigate the feasibility of hydrolyzing mixtures of energetics, particle size reduction, the formation of crystal (crystal growth) and the mixing of various hydrolysates.

NSWC will conduct bench scale studies to determine the heat of reaction of energetic materials at various alkaline concentrations by performing calorimetric studies. This information will be used to determine the amount of heat generated during reaction, which will provide a better ways to control reaction and eliminate reaction runaway.

Results and data generated from bench scale testing will be incorporated, as appropriate, into full-scale production process at Holston AAP.

## TECHNICAL APPROACH

The hydrolysis reaction will be carried out in a batch mode with samples collected at timed intervals. Based on experience, it is assumed that 5- to 10-hours of reactor residence time will be sufficient to achieve total destruction of the energetic material. The total destruction removal efficiency (DRE) goal is 99.999%. The following procedures will be used for executing this Test Program:

### Energetic Materials:

To fully characterize the hydrolysis reactor system for the destruction of energetic materials, seven (7) different energetic materials and mixtures were identified for processing in the pilot plant to support the scale-up and the design of the Pueblo and Lexington Bluegrass Chemical Agent Disposal Facilities. These are:

1. M1 - Nominal composition is 84% nitrocellulose, 9% dinitrotoluene, 5% dibutylphthalate, 1% diphenylamine, and 1% lead carbonate
2. M8 – Nominal composition is 52.15% nitrocellulose, 43% nitroglycerin, 3% diethylphthalate, 1.25% potassium nitrate, and 0.60% ethyl centralite
3. Tetrytol – Nominal composition is 70% Tetryl and 30% TNT
4. M28 grain– Nominal composition is 60% nitrocellulose, 23.8% nitroglycerin, 9.9% triacetin, 2.6% dimethylphthalate, and 2.0% lead stearate, and 1.7% 2-nitrodiphenylamine
5. Comp B – Nominal composition is 60% RDX (includes HMX percentages varying from 5-20%), 40% TNT, and plus 1% wax added
6. Comp B4 – Nominal composition is 59.75% RDX (includes HMX percentages varying from 5-20%), 39.75% TNT, and 0.50% calcium silicate
7. M28/Comp B4 (86/14 wt. %) based on their amount/ratio in the 115-mm Rocket, Chem Agent VX, M55

**Note:** Tetryl will NOT be hydrolyzed and characterized because of the limited supply of Tetryl and the health problems associated with handling this material. However, Tetrytol, which contains Tetryl as a component, will be fully characterized.

### Hydrolysis Reactor Operation:

For each of the above energetic materials to be fully hydrolyzed/destroyed, a pre-determined amount of alkaline solution can be calculated based on the weight of the energetic material as shown in Table 1 and Figure 2 will be added to the hydrolysis reactor.

- Alkaline Solution Strength – Individual and mixtures of energetic materials will be hydrolyzed using sodium hydroxide solution of 12, 20 and 25 wt. %.
- Quantity of Alkaline Solution -- Sodium hydroxide solution will be added in excess to insure total destruction of energetic materials.
- Alkaline Solution Temperature – The alkaline solution will be pre-heated to a maximum of 85 +/- 2 °C before the energetic materials are introduced into the reactor, and maintained at the temperature throughout the hydrolysis reaction.
- Rate of Addition -- A four-hour additional period with rates per hour as follows: 1<sup>st</sup> hour - 50 pounds per hour, 2<sup>nd</sup> hour - 100-pounds per hour; 3<sup>rd</sup> hour - 150-pounds per hour, 4<sup>th</sup> hour - 250-pounds per hour. Four-hour total of 500 pounds. This addition procedure will serve as the baseline for all tests.
- Agitator Speed – An optimum agitator speed will be selected to insure maximum mixing of energetics and soda solution to promote the reactions, and to avoid solids settling and foam formation.
- Residue -- At the conclusion of each run, the interior surfaces of the reactor will be visually inspected and the observations logged. Samples will be taken of any residue found within the reactor to determine the composition and to assess if operating parameters can be changed to minimize or eliminate formation of residue.
- Particle Size Distribution/Concentration -- Samples will be taken of the hydrolysate to characterize particle size and concentration.
- pH -- If the resulting pH of the hydrolysate is above 11, the pH will be adjusted using either phosphoric or sulfuric acid. If the hydrolysates are to be disposed of as waste, the pH adjustment will be performed with sulfuric acid; if the hydrolysates are to be used to support the SCWO technology demonstrations, the pH will be adjusted using phosphoric acid.

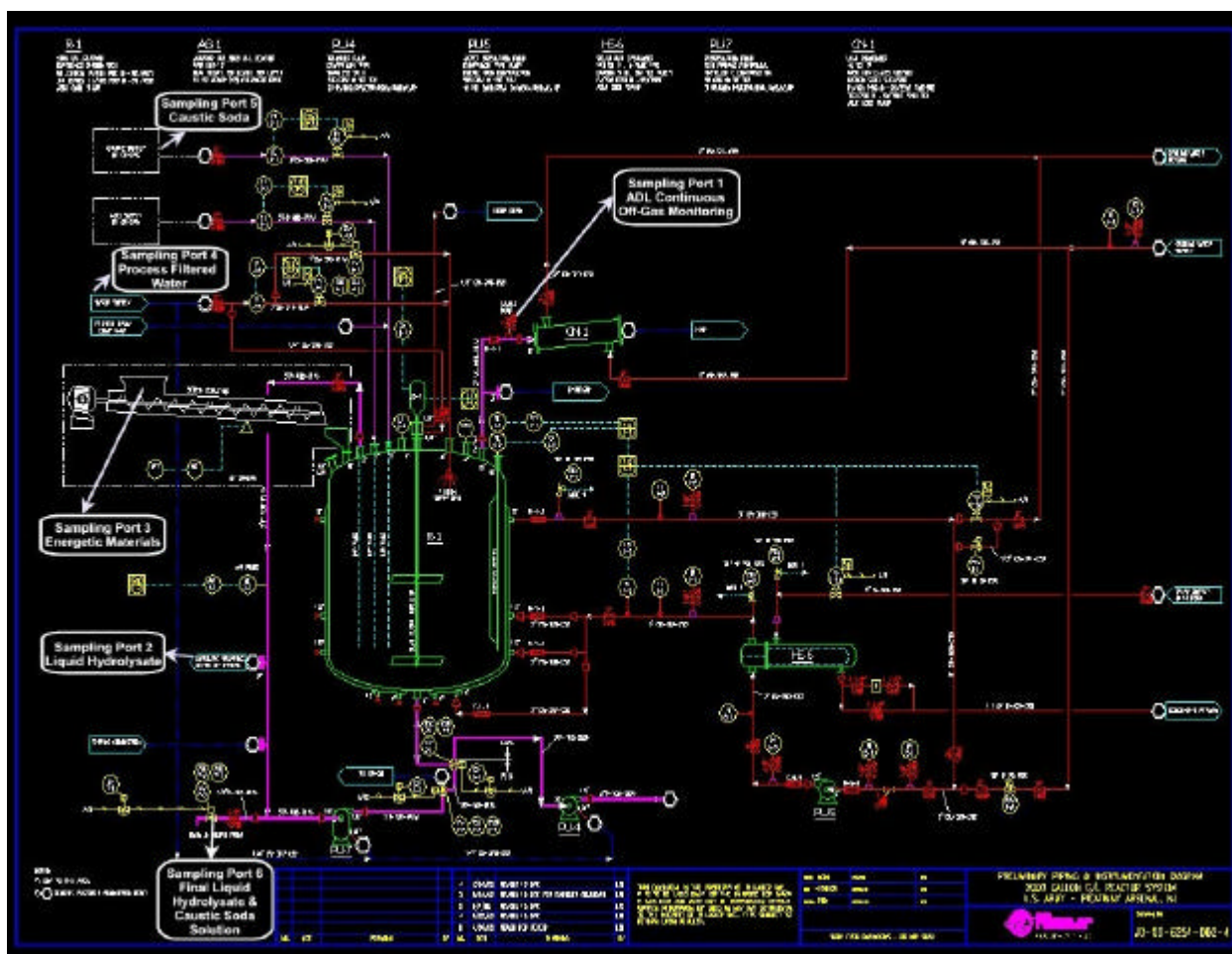
#### **Test Matrix:**

This test matrix will provide snapshot data, i.e., process operating parameters and samples analyses, from the proposed hydrolysis reactor system and fully characterize the hydrolysis reactions of energetic materials. The data and samples will be collected from various points within the process at various reactor residence times while operating at varying process operating conditions as shown in Figure 2. The test matrix shown in Table 1 will be used to study and characterize the pilot reactor system for the hydrolysis of energetic materials. The test plan includes a set of three (3) target process conditions at which the hydrolysis system will be monitored.

**Table 1., Test Plan for Hydrolysis Reactor**

<b>Test Condition</b>	<b>Reactor System Process Operating Parameters-Set Points</b>		
	<b>Hydrolysis Reactor Temperature (°C)</b>	<b>Reactor Residence Time (hr)</b>	<b>Alkaline Concentration (Wt. %)</b>
1	87 +/- 2 °C	10	12.00
2	87 +/- 2 °C	10	20.00
3	87 +/- 2 °C	10	25.00

A three factor/two level experimental design is used to develop the test plan, i.e.; the hydrolysis reactor will be controlled using three (3) operating parameters -- hydrolysis temperature, reactor residence time and alkaline concentration. The two levels represent the upper and lower limits for the three operating parameters. These limits will be determined based on experience and discussions with cognizant PM ACWA, contractor, TACOM-ARDEC, NSWC and LANL personnel.



**Figure 2., Hydrolysis Reactor System P&ID and Sampling Ports Identification**

**Note:** A minimum of three (3) test runs, one run conducted at each caustic strength cited in Table 3, will be performed for each energetic material. However, additional runs will be performed if necessary to verify data collected from previous runs.

### Data Collection:

Sample analysis of liquid hydrolysate, gaseous stream and solids will be collected and analyzed as specified in Tables 4-9. All process operating parameters and results of analysis for each set of conditions shown in Table 1 will be collected and recorded in the Sampling Log presented in Table 10. The test plan addresses liquid hydrolysate, gaseous and solids characterizations. The following analytes will be sampled and analyzed, as appropriate, to generate sufficient data to evaluate the process for destruction efficiency, efficacy and safety.

**Liquid Samples:** Liquid hydrolysate samples will be collected by RONA personnel per the Arthur D. Little, Inc. (ADL) specifications and guidance using the Intersystem Sampling System. The various sampling



ports on the reactor system are shown in Figure 2. The liquid hydrolysate samples will generally be collected at the interval times as shown in Table 2. Sample bottles supplied by ADL will be used to collect the hydrolysate

**Table 2. Sampling Time Intervals Matrix**

Minutes into Reaction, min		Sample #
	0	At completion of the Energetics Addition
Feed Addition – No Samples Collected	1 <sup>st</sup> hour	60
	2 <sup>nd</sup> hour	120
	3 <sup>rd</sup> hour	180
	4 <sup>th</sup> hour	240
Sampling every 15 min	5 <sup>th</sup> hour	255
		270
		285
		300
Sampling every 30 min	6 <sup>th</sup> hour	330
		360
	7 <sup>th</sup> hour	390
		420
	8 <sup>th</sup> hour	450
		480
	9 <sup>th</sup> hour	510
		540
Sampling every 60 min	10 <sup>th</sup> hour	600
	11 <sup>th</sup> hour	660
		14 at the end of reaction

samples. The bottles will be charged with pre-determined amount of 50 wt.% sulfuric acid and chilled to about 4 °C using ice to immediately quenched and stop the reaction from proceeding further, thereby insuring that the sample is as representative as possible of the actual time and temperature that it was collected at. The ratio of hydrolysate to acid will be based on achieving a target pH value between 6 and 8. The end-of-run liquid samples will be collected from a valve on the recycle loop on the reactor. Samples collected will be will be packaged per ADL specifications and sent to a specified test lab for analysis.

**Off-gas Effluents:** All off-gassing from the hydrolysis reaction will be monitored and sampled continuously using the ADL supplied sampling/analysis Field Trailer Control Room that will be connected directly into the off-gas port on the hydrolysis reactor cover. TRC, Inc. personnel will operate the Field Trailer complex.

**Sample Analysis:** To establish confidence limits on data gathered, all sample analysis will be conducted in triplicate. The three (3) individual results will be reported along with the average result including the range and the standard deviation. Using this procedure to collect data for all seven tests, 21 data points will be generated for each constituent evaluated throughout the study. This information will be used to determine the overall process variability of the data sets, which will then be used to establish confidence limits on the data. The more data collected, the more accurate and more detailed simulation model will be developed to assist in the scale-up and the design of the Pueblo and the Lexington Bluegrass Chemical Agent Disposal Facilities.

#### **Caustic Soda Requirement:**

Theoretically, the amount of Sodium Hydroxide (NaOH) is based on the number of nitramine, nitro, or nitrate ester on the energetic molecule; i.e., one mole of OH (NaOH) is required for each mole of nitramine, nitro, or nitrate ester to break the bond. An excess of 20% caustic is added to ensure that sufficient caustic is present

**Table 3: Caustic Soda (NaOH) Requirement**

<b>Energetic</b>	<b>Formula</b>	<b>MW</b>	<b>Wt. %</b>	<b>Num of nitramine, nitro, or nitrate ester</b>	<b>100% NaOH Lb.</b>	<b>6 wt.% NaOH Solution, Lb.</b>	<b>12 wt.% NaOH Solution, Lb.</b>	<b>20 wt.% NaOH Solution, Lb.</b>	<b>25 wt.% NaOH Solution Lb.</b>	<b>30 wt.% NaOH Solution, Lb.</b>
<b>Material</b>										
M1 Propellant			<b>500</b>							
Nitrocellulose (NC)		(272.39) <sub>n</sub>	84.00%	4	296.05	4934.10	2467.05	1480.23	1184.18	986.82
Dinitrooluene (DNT)	C <sub>7</sub> H <sub>6</sub> (NO <sub>2</sub> ) <sub>2</sub>	182.14	9.00%	2	23.72	395.30	197.65	118.59	94.87	79.06
Dibutylphthalate (DBP)	C <sub>16</sub> H <sub>22</sub> O <sub>4</sub>	278.35	5.00%							
Diphenylamine (DPA)	C <sub>12</sub> H <sub>11</sub> N	169.23	1.00%							
Lead Carbonate	CO <sub>3</sub> Pb	267.21	1.00%							
<b>TOTAL</b>			<b>100.00%</b>		<b>319.76</b>	<b>5329.40</b>	<b>2664.70</b>	<b>1598.82</b>	<b>1279.06</b>	<b>1065.88</b>
<b>Note:</b> For NC: only 3 ester groups on the molecule. Assume an extra mole due to possible change in nitration.										
M8 Propellant			<b>500</b>							
Nitrocellulose (NC)		(272.39) <sub>n</sub>	52.15%	4	183.80	3063.25	1531.63	918.98	735.18	612.65
Nitroglycerin (NG)	C <sub>3</sub> H <sub>5</sub> N <sub>3</sub> O <sub>9</sub>	227.09	43.00%	3	136.33	2272.23	1136.11	681.67	545.33	454.45
Diethylphthalate (DEP)	C <sub>12</sub> H <sub>14</sub> O <sub>4</sub>	222.24	3.00%							
Potassium Nitrate	KNO <sub>3</sub>	101.09	1.25%							
Ethyl Centralite	C <sub>17</sub> H <sub>20</sub> N <sub>2</sub> O	268.36	0.60%							
<b>TOTAL</b>			<b>100.00%</b>		<b>320.13</b>	<b>5335.48</b>	<b>2667.74</b>	<b>1600.64</b>	<b>1280.52</b>	<b>1067.10</b>
<b>Note:</b> For NC: only 3 ester groups on the molecule. Assume an extra mole due to possible change in nitration.										
Tetryl Explosive			<b>500</b>							
Tetryl	C <sub>7</sub> H <sub>5</sub> N(NO <sub>2</sub> ) <sub>4</sub>	287	100.00%	4	334.49	5574.91	2787.46	1672.47	1337.98	1114.98
<b>TOTAL</b>			<b>100.00%</b>		<b>334.49</b>	<b>5574.91</b>	<b>2787.46</b>	<b>1672.47</b>	<b>1337.98</b>	<b>1114.98</b>
Tetrytol Explosive			<b>500</b>							
Tetryl	C <sub>7</sub> H <sub>5</sub> N(NO <sub>2</sub> ) <sub>4</sub>	287	70.00%	4	234.15	3902.44	1951.22	1170.73	936.59	780.49
TNT	C <sub>7</sub> H <sub>5</sub> (NO <sub>2</sub> ) <sub>3</sub>	227	30.00%	3	95.15	1585.90	792.95	475.77	380.62	317.18
<b>TOTAL</b>			<b>100.00%</b>		<b>329.30</b>	<b>5488.34</b>	<b>2744.17</b>	<b>1646.50</b>	<b>1317.20</b>	<b>1097.67</b>

Material				or nitrate ester	Lb.	Solution, Lb.	Solution, Lb.	Solution, Lb.	Solution, Lb.	Solution, Lb.
Comp B/Comp B4 Explosive			500							
RDX	$C_3H_6N_3(NO_2)_3$	222.00	60.00%	3	194.59	3243.24	1621.62	972.97	778.38	648.65
TNT	$C_7H_5(NO_2)_3$	182.14	39.00%	3	154.17	2569.45	1284.73	770.84	616.67	513.89
HMX	$C_4H_8N_4(NO_2)_4$	296.00								
Ca Silicate	$CaSiO_3$	116.14								
Wax			1.00%							
<b>TOTAL</b>			<b>100.00%</b>		<b>348.76</b>	<b>5812.70</b>	<b>2906.35</b>	<b>1743.81</b>	<b>1395.05</b>	<b>1162.54</b>
M28 Propellant			500							
Nitrocellulose (NC)		(272.39) <sub>n</sub>	60.00%	4	211.46	3524.36	1762.18	1057.31	845.85	704.87
Nitroglycerin (NG)	$C_3H_5N_3O_9$	227.09	23.80%	3	75.46	1257.65	628.83	377.30	301.84	251.53
Triacetin	$C_9H_{14}O_6$	218.21	9.90%							
Dimethylphthalate (DMP)	$C_{10}H_{10}O_4$	194.19	2.60%							
Lead Stearate	$C_{36}H_{70}O_4Pb$	774.15	2.00%							
2-Nitrodiphenylamine (2NDPA)	$C_{12}H_{11}N$	169.23	1.70%							
<b>TOTAL</b>			<b>100.00%</b>		<b>286.92</b>	<b>4782.01</b>	<b>2391.00</b>	<b>1434.60</b>	<b>1147.68</b>	<b>956.40</b>
<b>Note:</b> For NC: only 3 ester groups on the molecule. Assume an extra mole due to possible change in nitration.										
M1/Tetrytol Mixture			500							
M1			92.00%		294.18	4902.99	2451.49	1470.90	1176.72	980.60
Tetrytol			8.00%		26.34	439.07	219.53	131.72	105.38	87.81
<b>TOTAL</b>			<b>100.00%</b>		<b>320.52</b>	<b>5342.05</b>	<b>2671.03</b>	<b>1602.62</b>	<b>1282.09</b>	<b>1068.41</b>
M8/Tetryl Mixture			500							
M8			69.00%		220.89	3681.495	1840.7475	1104.4485	883.56	736.30
Tetryl			31.00%		103.69	1728.20	864.10	518.46	414.77	345.64
<b>TOTAL</b>			<b>31.00%</b>		<b>324.58</b>	<b>5409.69</b>	<b>2704.85</b>	<b>1622.91</b>	<b>1298.33</b>	<b>1081.94</b>
M28/Comp B-B4 Mixture			500							
M28			70.00%		200.84	3347.41	1673.70	1004.22	803.38	669.48
Comp B-B4			30.00%		104.63	1743.81	871.90	523.14	418.51	348.76
<b>TOTAL</b>			<b>100.00%</b>		<b>305.47</b>	<b>5091.22</b>	<b>2545.61</b>	<b>1527.36</b>	<b>1221.89</b>	<b>1018.24</b>

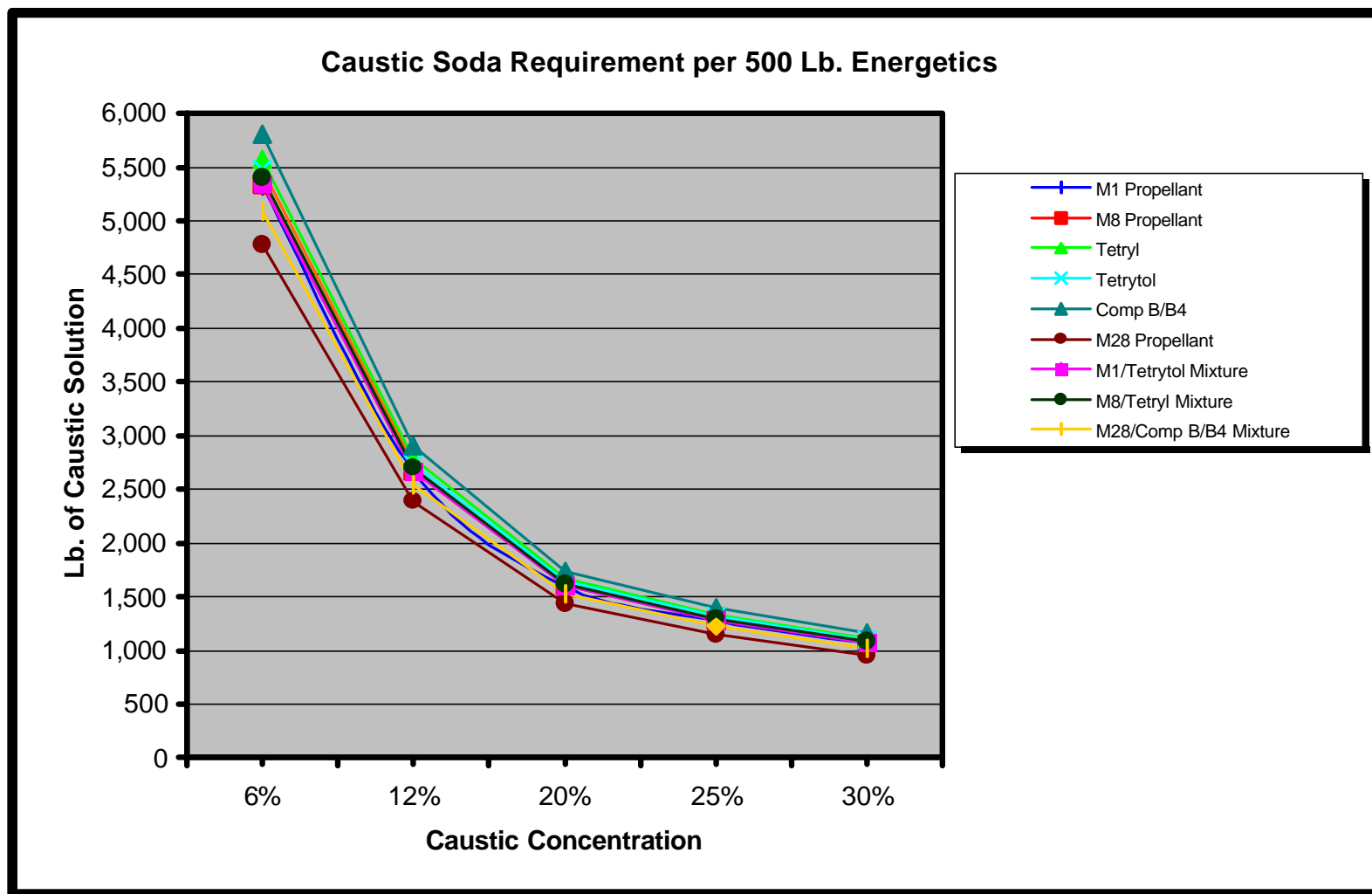


Figure 3., Caustic Soda Requirement per 500 Lb Energetic Materials

hydrolyze any energetic material generated as a by-product. The following formula is used to determine the amount of NaOH required for each test run.

$$\text{Mass of NaOH} = (\text{Mass of Explosive/Molecular Weight of Explosive}) * (\text{number of O-NO}_2, \text{N-NO}_2 \text{ or C-NO}_2 \text{ bonds}) * (40 \text{ g NaOH/mol}) * (1.2)$$

Table 1 and Figure 3 list the amount of caustic needed for each energetic material.

However, from an operational standpoint, the stoichiometric amount of caustic to carry the hydrolysis reaction to completion is **insufficient** to ensure the lower agitator blade assembly is completely immersed in the slurry, which is approximately 435 of the reactor working volume of 2,000 gallon. Therefore, it was decided that each test would be conducted with a starting point of 700-galons of caustic soda solution. Based on discussion with cognizant personnel at LANL, the additional caustic will not have a major effect on the hydrolysis of the energetics, rather the concentration which will be maintained at the desired level will have overall effect. The only effect will be the additional sulfuric acid needed to neutralize the hydrolysate.

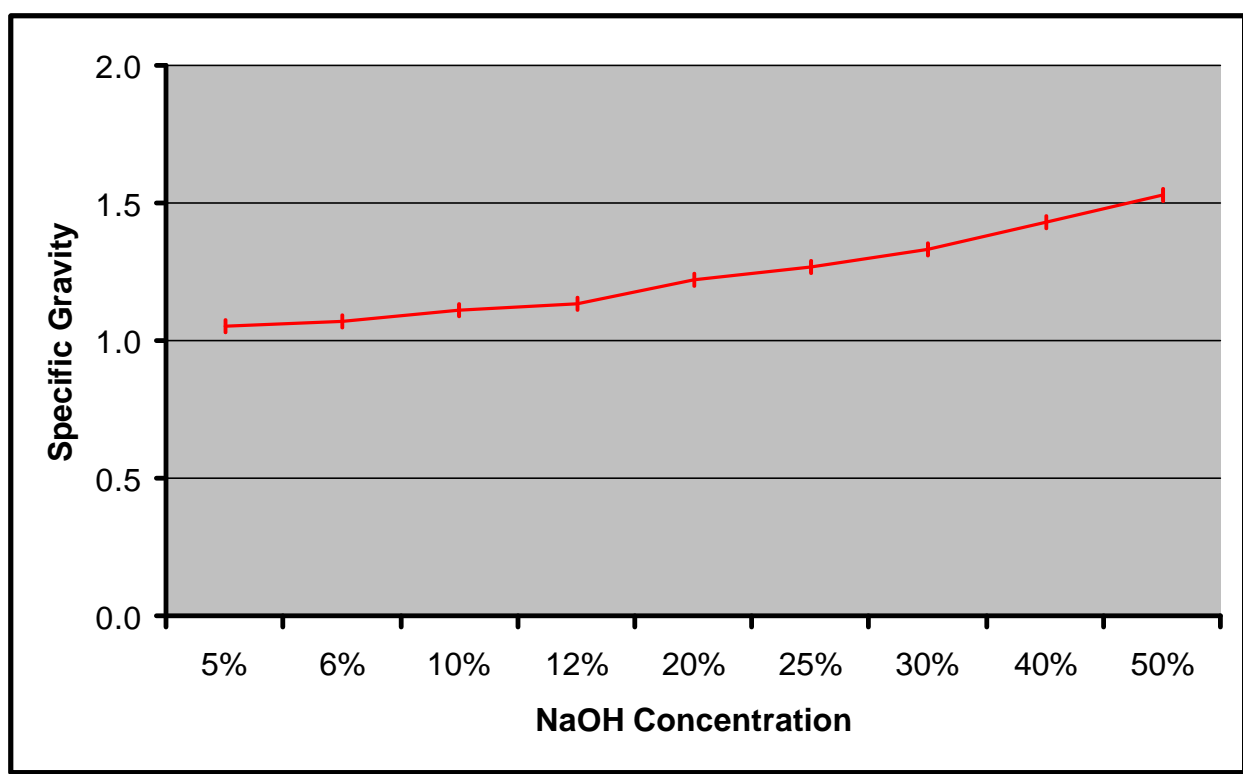


Figure 4. Specific Gravity of Sodium Hydroxide Solution

**Table 4, Additional Caustic Soda Requirement**

<b>500 Lb. - M1 Propellant</b>			
12%	2,665	282	1,118
20%	1,599	157	1,243
25%	1,279	125	1,275
<b>500 Lb. - M8 Propellant</b>			
12%	2,668	282	1,118
20%	1,601	157	1,243
25%	1,281	126	1,274
<b>500 Lb. - Tetryl Explosive</b>			
12%	2,787	295	1,105
20%	1,672	164	1,236
25%	1,338	131	1,269
<b>500 Lb. - Tetrytol Explosive</b>			
12%	2,744	290	1,110
20%	1,647	162	1,238
25%	1,317	129	1,271
<b>500 Lb. - Comp B / Comp B4 Explosive</b>			
12%	2,906	307	1,093
20%	1,744	171	1,229
25%	1,395	137	1,263
<b>500 Lb. - M28 Propellant</b>			
12%	2,391	253	1,347
20%	1,435	141	1,459
25%	1,148	113	1,287
<b>M28/Comp B - Comp B4 Energetic Mixture</b>			
12%	2,546	269	1,131
20%	1,527	150	1,250
25%	1,222	120	1,280

**Table 5. Demonstration Study Matrix – M1 Propellant Hydrolysis Processing**

Unit Operation	Test Objective	In Support of	Feed		Test Runs	Test Run Duration - Res. Time (hr)	Supporting Data Requirements	Process Streams		Test Location
			Energetic / Caustic	Quantity (lb.)				Type	Final Destination	
2,000 gal Hydrolysis Reactor System	<ul style="list-style-type: none"> <li>Hydrolyze M1 Propellant from the 105-mm cartridge, M60, HD in support of the scale-up and design of the Pueblo Disposal Facility</li> <li>Characterize gas, liquid, &amp; solid process streams from the energetics hydrolysis unit for selected chemical / constituents &amp; physical parameters, and the presence/absence of energetics, energetics hydrolysis products and other hazardous and toxic compounds</li> <li>Optimize process-operating parameters for the M1 propellant hydrolysis and obtain information applicable to completing the safety analyses, permitting, and NEPA documentation that would be required to implement base hydrolysis when production is scale-up.</li> </ul>	Pueblo Chemical Agent Disposal Facility	M1 Propellants / 12 wt.% NaOH	See Table 5.1	1	10	<ul style="list-style-type: none"> <li>Evaluate process effect on construction materials</li> <li>Confirm expected energetics reaction time</li> <li>Optimum Process Operating Parameters (Temp., Caustic Strength, Reactor Residence Time)</li> <li>Ratio of caustic consumed to feed</li> <li>Temperature vs. time</li> <li>Concentration vs. time</li> <li>Destruction Efficiency (DRE)</li> <li>Utility Requirements for operation and scale-up</li> </ul>	Liquid Hydrolysate	Ship to Authorized Waste Disposal Facility or to a Technology Provider, as required by the PM	Holston Army Ammunition Plant, Holston, TN
				See Table 5-1	1	10		Rinse Water		
			M1 Propellants / 20 wt.% NaOH	See Table 5-1	1	10		Decon Solution (Detergent)		
			M1 Propellants / 25 wt.% NaOH		1	10		Gas Reactor Headspace		
								Solids Filtered solids from liquid hydrolysate		

**Table 5-1. M1 Propellant Hydrolysis Schedule and Recipe**

<b>Energetic Material:</b>	<b>M1 Propellant</b>
<b>Hydrolysis Temperature:</b>	<b>87±2 °C (for all runs)</b>

Day	Incremental Rate Addition, Lb/ hr	Caustic Soda Heel, gal	Caustic Soda Concentration, Wt. %	Caustic Soda Needed, lb/lb Energetic	Caustic Soda Needed, gal	Caustic Soda Remaining, gal
<b>Run # 9</b> <b>February 28, 2001</b>	At Time 0, Add	700.00	12.00	5.33	0.00	700.00
	1 <sup>st</sup> hr: 50 lb/hr			266.47	28.16	671.84
	2 <sup>nd</sup> hr: 100 lb/hr			532.94	56.32	615.51
	3 <sup>rd</sup> hr: 150 lb/hr			799.41	84.49	531.03
	4 <sup>th</sup> hr: 200 lb/hr			1065.88	112.65	418.38
<b>Total Energetic Hydrolyzed</b>	<b>500.00</b>	<b>700.00</b>		<b>2664.70</b>	<b>281.62</b>	<b>418.38</b>
<b>Run # 10</b> <b>March 2, 2001</b>	At Time 0, Add	700.00	20.00	3.20	0.00	700.00
	1 <sup>st</sup> hr: 50 lb/hr			159.88	15.68	684.32
	2 <sup>nd</sup> hr: 100 lb/hr			319.76	31.37	652.95
	3 <sup>rd</sup> hr: 150 lb/hr			479.65	47.05	605.89
	4 <sup>th</sup> hr: 200 lb/hr			639.53	62.74	543.15
<b>Total Energetic Hydrolyzed</b>	<b>500.00</b>	<b>700.00</b>		<b>1598.82</b>	<b>156.85</b>	<b>543.15</b>
<b>Run # 11</b> <b>March 6, 2001</b>	At Time 0, Add	700.00	25.00	2.56	0.00	700.00
	1 <sup>st</sup> hr: 50 lb/hr			127.91	12.05	687.95
	2 <sup>nd</sup> hr: 100 lb/hr			255.81	24.11	663.84
	3 <sup>rd</sup> hr: 150 lb/hr			383.72	36.16	627.67
	4 <sup>th</sup> hr: 200 lb/hr			511.62	48.22	579.45
<b>Total Energetic Hydrolyzed</b>	<b>500.00</b>	<b>700.00</b>		<b>1279.06</b>	<b>120.55</b>	<b>579.45</b>



**Table 5-2. Demonstration Study Matrix – M1 Propellant Hydrolysis Sampling & Analysis**

Unit Oper- ation	Validation Sampling and Analysis						
	Sampling Location (include testing purpose)	Analytes (by location and Feed type)	# of Samples	Sample Collection Method	Sample Preservation Requirements	Sample Volume	Analytical Method
2,000 gal Hydr- ol- ysis Reac- tor Syste- m	Location: Port 3 – Figure 2  M1 Propellant Feed	a) Nitrocellulose (NC) b) Dinitrotoluene (DNT) c) Dibutylphthalate (DBP) d) Diphenylamine (DPA) e) Lead Carbonate	1 Sample/Feed	Take Sample	Quench immediately in a cold (~4 °C) Sulfuric Acid	100 gram	No analysis is needed. Assume M1 Composition is within Military Specifications
	Location: Port 5 or 6 – Figure 2  Caustic Feed NaOH Solution	a) Chloride b) Total Metals c) Carbonate	1 Sample/batch of NaOH Feed preparation	Take Sample	N/A	a) 250 ml b) 250 ml c) 50 ml	a) SW-846-9056 b) SW-846- 6010B/7470 c) ASTM E291-90
	Location: Port 4 – Figure 2  Filtered Process Water Feed	a) Chloride b) Total Metals c) Total Organic Carbon (TOC)	1 Sample/Feed	Take Sample	N/A	a) 250 ml b) 150 ml c) 10 ml	a) SW-846-9056 b) SW-846- 6010B/7470 c) SW-846-9060
	Location: Port 1 – Figure 2  Reactor Vessel off-gas	a) Nitrous Oxide (N <sub>2</sub> O) b) Nitrogen Oxide (NO <sub>x</sub> ) c) Nitrogen (N <sub>2</sub> ) d) Ammonia (NH <sub>3</sub> ) e) Carbon Monoxide (CO) f) Carbon Dioxide (CO <sub>2</sub> ) g) Hydrogen Cyanide (HCN) h) Aldehydes & ketones i) VOCs j) Total Organic Carbon (TOC)	Continuous Monitoring	a) CEMS b) In-Line c) Summa d) Impingers e) CEMS f) CEMS g) Impingers h) Impingers i) Summa j)	a) N/A b) N/A c) N/A d) 4°C e) 4°C f) N/A g) N/A h) 4°C i) N/A j) 4°C	a) N/A b) N/A/ c) 10 liter d) 100 ml e) N/A f) N/A g) 100 ml h) 100 ml i) 6 liter j) 100 ml	a) Method 7E b) NOISH 6600 c) TO-15/ASTM D1946 d) Method 26/CTM- 027 e) Method 0010 f) Method 3A g) Method 26/CARB 426 h) Method 0011/8315A i) TO-15 j) SW-846-9060
	Location: Port 2 – Figure 2  Reactor Vessel Liquid/Solid - Hydrolysate	a) Energetic Content b) NC c) DNT d) NO <sub>2</sub> /NO <sub>3</sub> e) Cyanide by-Product f) Picric Acid – Picrate Salts (Sodium, etc.) g) Total Organic Carbon (TOC) h) Total Inorganic Carbon i) Total Metals (Pb)	1 Sample/Time (min)  See Table 2 (Sampling Interval Matrix)	Take Sample via Remote Sampling System (Intersystem)	a) 4°C b) 4°C c) 4°C d) 4°C e) 4°C f) 4°C g) 4°C & pH <2 h) 4°C i) 4°C & pH<2	a) 50 ml b) 10 ml c) 10 ml d) 10 ml e) 10 ml f) 10 ml g) 50 ml h) 10 ml i)	a) DSC b) GPC/FTIR c) SW-846-8330 d) SW-846-9056 e) SW-846- 9010/9014 f) GPC/FTIR g) SW-846-9060 h) SW-846-9060 i) SW-846- 6010/7470

**Table 6. Demonstration Study Matrix – M8 Propellant Hydrolysis Processing**

Unit Operation	Test Objective	In Support of	Feed		Test Runs	Test Run Duration - Res. Time (hr)	Supporting Data Requirements	Process Streams		Test Location
			Energetic / Caustic	Quantity (lb.)				Type	Final Destination	
2,000 gal Hydrolysis Reactor System	<ul style="list-style-type: none"> <li>Hydrolyze M8 Propellant from the 4.2-in cartridge, HD &amp; HT, M2 &amp; M2A1 in support of the scale-up and design of the Pueblo Disposal Facility</li> <li>Characterize gas, liquid, &amp; solid process streams from the energetics hydrolysis unit for selected chemical/constituents &amp; physical parameters, and the presence/absence of energetics, energetics hydrolysis products and other hazardous and toxic compounds</li> <li>Optimize process-operating parameters for the M8 propellant hydrolysis and obtain information applicable to completing the safety analyses, permitting, and NEPA documentation that would be required to implement base hydrolysis when production is scale-up.</li> </ul>	Pueblo Chemical Agent Disposal Facility	M8 Propellants / 12 wt.% NaOH	See Table 6-1	1	10	<ul style="list-style-type: none"> <li>Evaluate process effect on construction materials</li> <li>Confirm expected energetics reaction time</li> <li>Optimum Process Operating Parameters (Temp., Caustic Strength, Reactor Residence Time)</li> <li>Ratio of caustic consumed to feed</li> <li>Temperature vs. time</li> <li>Concentration vs. time</li> <li>Destruction Efficiency (DRE)</li> <li>Utility Requirements for operation and scale-up</li> </ul>	Liquid Hydrolysate	Ship to Authorized Waste Disposal Facility or to a Technology Provider, as required by the PM	Holston Army Ammunition Plant, Holston, TN
					1	10		Rinse Water		
			M8 Propellants / 20 wt.% NaOH	See Table 6-1	1	10		Decon Solution (Detergent)		
			M8 Propellants / 25 wt.% NaOH	See Table 6-1	1	10		Gas Reactor Headspace		
								Solids Filtered solids from liquid hydrolysate		

Table 6-1. M8 Propellant Hydrolysis Schedule and Recipe

**Energetic Material:** M8 Propellant

**Hydrolysis Temperature:** 87±2 °C (for all runs)

Day	Incremental Rate Addition, Lb/ hr	Caustic Soda Heel, gal	Caustic Soda Concentration, Wt. %	Caustic Soda Needed, lb/lb Energetic	Caustic Soda Needed, gal	Caustic Soda Remaining, gal
<b>Run # 18</b> <b>April 3, 2001</b>	At Time 0, Add	700.00	12.00	5.34	0.00	700.00
	1 <sup>st</sup> hr: 50 lb/hr			266.77	28.19	671.81
	2 <sup>nd</sup> hr: 100 lb/hr			533.55	56.39	615.42
	3 <sup>rd</sup> hr: 150 lb/hr			800.32	84.58	530.83
	4 <sup>th</sup> hr: 200 lb/hr			1067.10	112.78	418.06
<b>Total Energetic Hydrolyzed</b>	<b>500.00</b>	<b>700.00</b>		<b>2667.74</b>	<b>281.94</b>	<b>418.06</b>
<b>Run # 19</b> <b>April 12, 2001</b>	At Time 0, Add	700.00	20.00	3.20	0.00	700.00
	1 <sup>st</sup> hr: 50 lb/hr			160.06	15.70	684.30
	2 <sup>nd</sup> hr: 100 lb/hr			320.13	31.40	652.89
	3 <sup>rd</sup> hr: 150 lb/hr			480.19	47.11	605.79
	4 <sup>th</sup> hr: 200 lb/hr			640.26	62.81	542.98
<b>Total Energetic Hydrolyzed</b>	<b>500.00</b>	<b>700.00</b>		<b>1600.64</b>	<b>157.02</b>	<b>542.98</b>
<b>Run # 20</b> <b>April 18, 2001</b>	At Time 0, Add	700.00	25.00	2.56	0.00	700.00
	1 <sup>st</sup> hr: 50 lb/hr			128.05	12.07	687.93
	2 <sup>nd</sup> hr: 100 lb/hr			256.10	24.14	663.80
	3 <sup>rd</sup> hr: 150 lb/hr			384.16	36.20	627.59
	4 <sup>th</sup> hr: 200 lb/hr			512.21	48.27	579.32
<b>Total Energetic Hydrolyzed</b>	<b>500.00</b>	<b>700.00</b>		<b>1280.52</b>	<b>120.68</b>	<b>579.32</b>

**Table 6-2. Demonstration Study Matrix – M8 Propellant Hydrolysis Sampling & Analysis**

Unit Operation	Validation Sampling and Analysis						
	Sampling Location (include testing purpose)	Analytes (by location and feed type)	# of Samples	Sample Collection Method	Sample Preservation Requirements	Sample Volume	Analytical Method
2,000 gal Hydrolysis Reactor System	Location: Port 3 – Figure 2  M8 Propellant Feed	a) Nitrocellulose (NC) b) Nitroglycerin (NG) c) Diethylphthalate (DEP) d) Potassium Nitrate e) Ethyl Centralite (EC)	1 Sample/Feed	Take Sample	Quench immediately in a cold (~4 °C) Sulfuric Acid	100 gram	No analysis is needed. Assume M8 Composition is within Military Specifications
	Location: Port 5 or 6 – Figure 2  Caustic Feed NaOH Solution	a) Chloride b) Total Metals c) Carbonate	1 sample/batch of NaOH feed preparation	Take Sample	N/A	a) 250 ml b) 250 ml c) 50 ml	a) SW-846-9056 b) SW-846-6010B/7470 c) ASTM E291-90
	Location: Port 4 – Figure 2  Filtered Process Water Feed	a) Chloride b) Total Metals c) Total organic Carbon (TOC)	1 Sample/Feed	Take Sample	N/A	a) 250 ml b) 150 ml c) 10 ml	a) SW-846-9056 b) SW-846-6010B/7470 c) SW-846-9060
	Location: Port 1 - Figure 2  Reactor Vessel off-gas	a) Nitrous Oxide (N <sub>2</sub> O) b) Nitrogen Oxide (NO <sub>x</sub> ) c) Nitrogen (N <sub>2</sub> ) d) Ammonia (NH <sub>3</sub> ) e) Carbon Monoxide (CO) f) Carbon Dioxide (CO <sub>2</sub> ) g) Hydrogen Cyanide (HCN) h) Aldehydes & ketones i) VOCs j) Total Organic Carbon (TOC)	Continuous Monitoring	a) CEMS b) In-Line c) Summa d) Impingers e) CEMS f) CEMS g) Impingers h) Impingers i) Summa j)	a) N/A b) N/A c) N/A d) 4°C e) 4°C f) N/A g) N/A h) 4°C i) N/A j) 4°C	a) N/A b) N/A/ c) 10 liter d) 100 ml e) N/A f) N/A g) 100 ml h) 100 ml i) 6 liter j) 100 ml	a) Method 7E b) NOISH 6600 c) TO-15/ASTM D1946 d) Method 26/CTM-027 e) Method 0010 f) Method 3A g) Method 26/CARB 426 h) Method 0011/8315A i) TO-15 j) SW-846-9060
	Location: Port 2 – Figure 2  Reactor Vessel Liquid/Solids - Hydrolysate	a) Energetic Content b) NC c) NG d) NO <sub>2</sub> /NO <sub>3</sub> e) Cyanide by-Product f) Picric Acid – Picrate Salts (Sodium, etc.) g) Total Organic Carbon (TOC) h) Total Inorganic Carbon i) Total Metals	1 Sample/Time (min)  See Table 2 (Sampling Interval Matrix)	Take Sample via Remote Sampling System (Intersystem)	a) 4°C b) 4°C c) 4°C d) 4°C e) 4°C f) 4°C g) 4°C h) 4°C i) 4°C & pH<2	a) 50 ml b) 10 ml c) 1 ml d) 10 ml e) 10 ml f) TBD g) 10 ml h) 10 ml i) 10 ml	a) DSC b) GPC/FTIR c) SW-846-8332 d) SW-846-9056 e) SW-846-9010B/9014 f) TBD g) SW-846-9060 h) SW-846-9060 Mod. i) SW-846-6010/7470

**Table 7. Demonstration Study Matrix – Tetrytol Explosive Hydrolysis Processing**

Unit Operation	Test Objective	In Support of	Feed		Test Runs	Test Run Duration - Res. Time (hr)	Supporting Data Requirements	Process Streams		Test Location
			Energetic / Caustic	Quantity (lb.)				Type	Final Destination	
2,000 gal Hydrolysis Reactor System	<ul style="list-style-type: none"> <li>Hydrolyze Tetrytol Explosive from the 155-mm HD, M110 &amp; M104 and from the 105-mm, HD, M60 cartridge in support of the scale-up and design of the Pueblo &amp; the Lexington Bluegrass Disposal Facilities</li> <li>Characterize gas, liquid, &amp; solid process streams from the energetics hydrolysis unit for selected chemical / constituents &amp; physical parameters, and the presence/absence of energetics, energetics hydrolysis products and other hazardous and toxic compounds</li> <li>Optimize process-operating parameters for the Tetrytol explosive hydrolysis and obtain information applicable to completing the safety analyses, permitting, and NEPA documentation that would be required to implement base hydrolysis when production is scale-up.</li> </ul>	Lexington Bluegrass Chemical Agent Disposal Facility	Tetrytol Explosive / 12 wt.% NaOH	See Table 7-1	1	10	<ul style="list-style-type: none"> <li>Evaluate process effect on construction materials</li> </ul>	Liquid Hydrolysate	Ship to Authorized Waste Disposal Facility or to a Technology Provider, as required by the PM	Holston Army Ammunition Plant, Holston, TN
			Tetrytol Explosive / 20 wt.% NaOH	See Table 7-1	1	10	<ul style="list-style-type: none"> <li>Confirm expected energetics reaction time</li> </ul>	Rinse Water		
			Tetrytol Explosive / 25 wt.% NaOH	See Table 7-1	1	10	<ul style="list-style-type: none"> <li>Optimum Process Operating Parameters (Temp., Caustic Strength, Reactor Residence Time)</li> </ul>	Decon Solution (Detergent)		
					1	10	<ul style="list-style-type: none"> <li>Ratio of caustic consumed to feed</li> <li>Temperature vs. time</li> <li>Concentration vs. time</li> <li>Destruction Efficiency (DRE)</li> <li>Utility Requirements for operation and scale-up</li> </ul>	Gas Reactor Headspace  Solids Filtered solids from liquid hydrolysate		

Table 7-1. Tetrytol Explosive Hydrolysis Schedule and Recipe

<b>Energetic Material:</b>	<b>Tetrytol Explosive</b>
<b>Hydrolysis Temperature:</b>	<b>87±2 °C (for all runs)</b>

Day	Incremental Rate Addition, Lb/ hr	Caustic Soda Heel, gal	Caustic Soda Concentration, Wt. %	Caustic Soda Needed, lb/lb Energetic	Caustic Soda Needed, gal	Caustic Soda Remaining, gal
<b>Run # 15</b>	At Time 0, Add	700.00	12.00	5.49	0.00	700.00
<b>March 26, 2001</b>	1 <sup>st</sup> hr: 50 lb/hr			274.42	29.00	671.00
	2 <sup>nd</sup> hr: 100 lb/hr			548.83	58.00	612.99
	4 <sup>th</sup> hr: 200 lb/hr			1097.67	116.01	496.99
<b>Total Energetic Hydrolyzed</b>	<b>350.00</b>	<b>700.00</b>		<b>1920.92</b>	<b>203.01</b>	<b>496.99</b>
<b>Run # 16</b>	At Time 0, Add	700.00	20.00	3.29	0.00	700.00
<b>March 28, 2001</b>	1 <sup>st</sup> hr: 50 lb/hr			164.65	16.15	683.85
	2 <sup>nd</sup> hr: 100 lb/hr			329.30	32.30	651.54
	4 <sup>th</sup> hr: 200 lb/hr			658.60	64.61	586.93
<b>Total Energetic Hydrolyzed</b>	<b>350.00</b>	<b>700.00</b>		<b>1152.55</b>	<b>113.07</b>	<b>586.93</b>
<b>Run # 17</b>	At Time 0, Add	700.00	25.00	2.63	0.00	700.00
<b>March 30, 2001</b>	1 <sup>st</sup> hr: 50 lb/hr			131.72	12.41	687.59
	2 <sup>nd</sup> hr: 100 lb/hr			263.44	24.83	662.76
	4 <sup>th</sup> hr: 200 lb/hr			526.88	49.66	613.10
<b>Total Energetic Hydrolyzed</b>	<b>350.00</b>	<b>700.00</b>		<b>922.04</b>	<b>86.90</b>	<b>613.10</b>

**Table 7-2. Demonstration Study Matrix – Tetrytol Explosive Hydrolysis Sampling & Analysis**

Unit Operation	Validation Sampling and Analysis						
	Sampling Location (include testing purpose)	Analytes (by location and feed type)	# of Samples	Sample Collection Method	Sample Preservation Requirements	Sample Volume	Analytical Method
2,000 gal Hydrolysis Reactor System	Location: Port 3 – Figure 2 Tetryl Explosive Feed	a) Tetryl, TNT	1 Sample/Feed	Take Sample	Quench immediately in a cold (~4 °C) Sulfuric Acid	100 gram	No analysis is needed. Assume Tetrytol Composition is within Military Specifications
	Location: Port 5 or 6 – Figure 2 Caustic Feed NaOH Solution	a) Chloride b) Total Metals c) Carbonate	1 Sample/batch of NaOH feed preparation	Take Sample	N/A	a) 250 ml b) 250 ml c) 50 ml	a) SW-846-9056 b) SW-846-6010B/7470 c) ASTM E291-90
	Location: Port 4 – Figure 2 Filtered Process Water Feed	a) Chloride b) Total Metals c) Total organic Carbon (TOC)	1 Sample/Feed	Take Sample	N/A	a) 250 ml b) 150 ml c) 10 ml	a) SW-846-9056 b) SW-846-6010B/7470 c) SW-846-9060
	Location: Port 1 – Figure 2 Reactor Vessel off-gas	a) Nitrous Oxide (N <sub>2</sub> O) b) Nitrogen Oxide (NO <sub>x</sub> ) c) Nitrogen (N <sub>2</sub> ) d) Ammonia (NH <sub>3</sub> ) e) Carbon Monoxide (CO) f) Carbon Dioxide (CO <sub>2</sub> ) g) Hydrogen Cyanide (HCN) h) Aldehydes & ketones i) VOCs j) Total Hydrocarbon (TOC)	Continuous Monitoring	a) CEMS b) In-Line c) Summa d) Impingers e) CEMS f) CEMS g) Impingers h) Impingers i) Summa j)	a) N/A b) N/A c) N/A d) 4°C e) 4°C f) N/A g) N/A h) 4°C i) N/A j) 4°C	a) N/A b) N/A/ c) 10 liter d) 100 ml e) N/A f) N/A g) 100 ml h) 100 ml i) 6 liter j) 100 ml	a) Method 7E b) NOISH 6600 c) TO-15/ASTM D1946 d) Method 26/CTM-027 e) Method 0010 f) Method 3A g) Method 26/CARB 426 h) Method 0011/8315A i) TO-15 j) SW-846-9060
	Location: Port 2 – Figure 2 Reactor Vessel Liquid/Solid - Hydrolysate	a) Energetic Content b) Tetryl, TNT c) NO <sub>2</sub> /NO <sub>3</sub> d) Cyanide by-Product e) Picric Acid – Picrate Salts (Sodium, etc.) f) Total Organic Carbon (TOC) g) Total Inorganic Carbon h) Total Metals	1 Sample/Time (min) See Table 2 (Sampling Interval Matrix)	Take Sample via Remote Sampling System (Intersystem)	a) 4°C b) 4°C c) 4°C d) 4°C e) 4°C f) 4°C g) 4°C h) 4°C & pH<2	a) 50 ml b) 10 ml c) 10 ml d) 10 ml e) TBD f) 10 ml g) 10 ml h) 10 ml	a) DSC b) SW-846-8330 c) SW-846-9056 d) SW-846-9010B/914 e) TBD f) SW-846-9060 g) SW-846-9060 Mod. h) SW-846-6010/7470

**Table 8. Demonstration Study Matrix – Comp B/Comp B4 Explosive Hydrolysis Processing**

Unit Operation	Test Objective	In Support of	Feed		Test Runs	Test Run Duration - Res. Time (hr)	Supporting Data Requirements	Process Streams		Test Location
			Energetic / Caustic	Quantity (lb.)				Type	Final Destination	
2,000 gal Hydrolysis Reactor System	<ul style="list-style-type: none"> <li>Hydrolyze Comp B/Comp B4 Explosive from the 8-in GB, M426 Projectile, 155-mm HD, M110 &amp; VX M121/A1 Projectile, 115-mm GB, M55Rocket, &amp; M56 Rocket Warhead in support of the scale-up and design of the Lexington Bluegrass Disposal Facility</li> <li>Characterize gas, liquid, &amp; solid process streams from the energetics hydrolysis unit for selected chemical / constituents &amp; physical parameters, and the presence/absence of energetics, energetics hydrolysis products and other hazardous and toxic compounds</li> <li>Optimize process operating parameters for the Comp B/Comp B4 explosives hydrolysis and obtain information applicable to completing the safety analyses, permitting, and NEPA documentation that would be required to implement base hydrolysis when production is scale-up.</li> </ul>	Lexington Bluegrass Chemical Agent Disposal Facility	Comp B/Comp B4 Explosive / 12 wt.% NaOH	See Table 8-1	1	10	<ul style="list-style-type: none"> <li>Evaluate process effect on construction materials</li> </ul>	Liquid Hydrolysate	Ship to Authorized Waste Disposal Facility or to a Technology Provider, as required by the PM	Holston Army Ammunition Plant, Holston, TN
			Comp B/Comp B4 Explosive / 20 wt.% NaOH	See Table 8-1	1	10	<ul style="list-style-type: none"> <li>Confirm expected energetics reaction time</li> </ul>	Rinse Water		
			Comp B/Comp B4 Explosive / 25 wt.% NaOH	See Table 8-1	1	10	<ul style="list-style-type: none"> <li>Optimum Process Operating Parameters (Temp., Caustic Strength, Reactor Residence Time)</li> </ul>	Decon Solution (Detergent)		
					1	10	<ul style="list-style-type: none"> <li>Ratio of caustic consumed to feed</li> <li>Temperature vs. time</li> <li>Concentration vs. time</li> <li>Destruction Efficiency (DRE)</li> <li>Utility Requirements for operation and scale-up</li> </ul>	Gas Reactor Headspace		
								Solids Filtered solids from liquid hydrolysate		



Energetic Material:	Comp B4
Hydrolysis Temperature:	87±2 °C (for all runs)

Day	Incremental Rate Addition, Lb/ hr	Caustic Soda Heel, gal	Caustic Soda Concentration, Wt. %	Caustic Soda Needed, lb/lb Energetic	Caustic Soda Needed, gal	Caustic Soda Remaining, gal
<b>Run # 6</b> <b>February 15, 2001</b>	At Time 0, Add	700.00	12.00	5.81	0.00	700.00
	1 <sup>st</sup> hr: 50 lb/hr			290.50	30.70	669.30
	2 <sup>nd</sup> hr: 100 lb/hr			581.00	61.40	607.89
	3 <sup>rd</sup> hr: 150 lb/hr			871.50	92.11	515.79
	4 <sup>th</sup> hr: 200 lb/hr			1162.00	122.81	392.98
<b>Total Energetic Hydrolyzed</b>	<b>500.00</b>	<b>700.00</b>		<b>2905.00</b>	<b>307.02</b>	<b>392.98</b>
<b>Run # 7</b> <b>February 20, 2001</b>	At Time 0, Add	700.00	20.00	3.49	0.00	700.00
	1 <sup>st</sup> hr: 50 lb/hr			174.50	17.12	682.88
	2 <sup>nd</sup> hr: 100 lb/hr			349.00	34.24	648.64
	3 <sup>rd</sup> hr: 150 lb/hr			523.50	51.36	597.29
	4 <sup>th</sup> hr: 200 lb/hr			698.00	68.47	528.81
<b>Total Energetic Hydrolyzed</b>	<b>500.00</b>	<b>700.00</b>		<b>1745.00</b>	<b>171.19</b>	<b>528.81</b>
<b>Run # 8</b> <b>February 26, 2001</b>	At Time 0, Add	700.00	25.00	2.79	0.00	700.00
	1 <sup>st</sup> hr: 50 lb/hr			139.50	13.15	686.85
	2 <sup>nd</sup> hr: 100 lb/hr			279.00	26.29	660.56
	3 <sup>rd</sup> hr: 150 lb/hr			418.50	39.44	621.12
	4 <sup>th</sup> hr: 200 lb/hr			558.00	52.59	568.53
<b>Total Energetic Hydrolyzed</b>	<b>500.00</b>	<b>700.00</b>		<b>1395.00</b>	<b>131.47</b>	<b>568.53</b>

**Table 8-2. Demonstration Study Matrix – Comp B/Comp B4 Explosive Hydrolysis Sampling & Analysis**

Unit Operation	Validation Sampling and Analysis						
	Sampling Location (include testing purpose)	Analytes (by location and feed type)	# of Samples	Sample Collection Method	Sample Preservation Requirements	Sample Volume	Analytical Method
2,000 gal Hydrolysis Reactor System	Location: Port 3 – Figure 2 Comp B Explosive Feed	RDX, HMX TNT Wax Ca Silicate	1 Sample/Feed	Take Sample	Quench immediately in a cold (~4 °C) Sulfuric Acid	100 gram	No analysis is needed. Assume Comp B/B4 Composition is within Military Specifications
	Location: Port 5 or 6 – Figure 2 Caustic Feed NaOH Solution	Chloride Total Metals Carbonate	1 sample/batch of NaOH feed preparation	Take Sample	N/A	250 ml 250 ml 50 ml	SW-846-9056 SW-846-6010B/7470 ASTM E291-90
	Location: Port 4 – Figure 2 Filtered Process Water Feed	Chloride Total Metals Total organic Carbon (TOC)	1 Sample/Feed	Take Sample	N/A	50 ml 50 ml 0 ml	SW-846-9056 SW-846-6010B/7470 SW-846-9060
	Location: Port 1 – Figure 2 Reactor Vessel off-gas	Nitrous Oxide (N <sub>2</sub> O) Nitrogen Oxide (NO <sub>x</sub> ) Nitrogen (N <sub>2</sub> ) Ammonia (NH <sub>3</sub> ) Carbon Monoxide (CO) Carbon Dioxide (CO <sub>2</sub> ) Hydrogen Cyanide (HCN) Aldehydes & ketones VOCs Total Hydrocarbon (TOC)	Continuous Monitoring	CEMS On-Line Summa Impingers CEMS CEMS Impingers Impingers Summa	N/A N/A N/A °C °C N/A N/A °C °C N/A °C	N/A N/A/ 0 liter 00 ml N/A N/A 00 ml 00 ml 00 ml liter 00 ml	Method 7E EPA 8210-1015H 6600 EPA 8210-1015/ASTM D1946 Method 26/CTM-027 Method 0010 Method 3A Method 26/CARB 426 Method 0011/8315A EPA 8210-1015 SW-846-9060
	Location: Port 2 – Figure 2 Reactor Vessel Liquid/Solids - Hydrolysate	Energetic Content RDX, HMX, TNT NO <sub>2</sub> /NO <sub>3</sub> Cyanide By-product Picric Acid – Picrate Salts Sodium, etc.) Total Organic Carbon (TOC) Total Inorganic Carbon Total Metals	1 Sample/Time (min)  See Table 2 Sampling Interval Matrix)	Take Sample via Remote Sampling System (Intersystem)	4°C 4°C 4°C 4°C 4°C 4°C 4°C & pH<2	50 ml 10 ml 10 ml 50 ml TBD 10 ml 10 ml 10 ml	DSC SW-846-8330 / EPA 8210-1015H GC/ECD SW-846-9056 SW-846-6010B/9014 TBD SW-846-9060 SW-846-9060 Mod. SW-846-6010/7470

**Table 9. Demonstration Study Matrix – M28 Propellant Hydrolysis Processing**

Unit Operation	Test Objective	In Support of	Feed		Test Runs	Test Run Duration - Res. Time (hr)	Supporting Data Requirements	Process Streams		Test Location
			Energetic / Caustic	Quantity (lb.)				Type	Final Destination	
2,000 gal Hydrolysis Reactor System	<ul style="list-style-type: none"> <li>Hydrolyze M28 Propellant from the 115-mm, GB, M55 &amp; M56 projectile and the 115-mm, GB, M56 &amp; VX, M55 &amp; M56 Rocket &amp; Rocket Warhead in support of the scale-up and design of the Lexington Bluegrass Disposal Facility</li> <li>Characterize gas, liquid, &amp; solid process streams from the energetics hydrolysis unit for selected chemical / constituents &amp; physical parameters, and the presence/absence of energetics, energetics hydrolysis products and other hazardous and toxic compounds</li> <li>Optimize process-operating parameters for the M28 propellant hydrolysis and obtain information applicable to completing the safety analyses, permitting, and NEPA documentation that would be required to implement base hydrolysis when production is scale-up.</li> </ul>	Lexington Bluegrass Chemical Agent Disposal Facility	M28 Propellants / 12 wt.% NaOH	See Table 9-1	1	10	<ul style="list-style-type: none"> <li>Evaluate process effect on construction materials</li> </ul>	Liquid Hydrolysate	Ship to Authorized Waste Disposal Facility or to a Technology Provider, as required by the PM	Holston Army Ammunition Plant, Holston, TN
			M28 Propellants / 20 wt.% NaOH	See Table 9-1	1	10	<ul style="list-style-type: none"> <li>Confirm expected energetics reaction time</li> </ul>	Rinse Water		
			M28 Propellants / 25 wt.% NaOH	See Table 9-1	1	10	<ul style="list-style-type: none"> <li>Optimum Process Operating Parameters (Temp., Caustic Strength, Reactor Residence Time)</li> </ul>	Decon Solution (Detergent)		
					1	10	<ul style="list-style-type: none"> <li>Ratio of caustic consumed to feed</li> <li>Temperature vs. time</li> <li>Concentration vs. time</li> <li>Destruction Efficiency (DRE)</li> <li>Utility Requirements for operation and scale-up</li> </ul>	Gas Reactor Headspace  Solids Filtered solids from liquid hydrolysate		



**Table 9-1. M28 Propellant Hydrolysis Schedule and Recipe**

<b>Energetic Material:</b>	<b>M28 Propellant (Unleaded)</b>
<b>Hydrolysis Temperature:</b>	<b>87±2 °C (for all runs)</b>

<b>Day</b>	<b>Incremental Rate Addition, Lb/ hr</b>	<b>Caustic Soda Heel, gal</b>	<b>Caustic Soda Concentration, Wt. %</b>	<b>Caustic Soda Needed, lb/lb Energetic</b>	<b>Caustic Soda Needed, gal</b>	<b>Caustic Soda Remaining, gal</b>
<b>Run # 12</b>	At Time 0, Add	700.00	12.00	4.78	0.00	700.00
<b>March 9, 2001</b>	1 <sup>st</sup> hr: 50 lb/hr			239.10	25.27	674.73
	2 <sup>nd</sup> hr: 100 lb/hr			478.20	50.54	624.19
	3 <sup>rd</sup> hr: 150 lb/hr			717.30	75.81	548.38
	4 <sup>th</sup> hr: 200 lb/hr			956.40	101.08	447.30
<b>Total Energetic Hydrolyzed</b>	<b>500.00</b>	<b>700.00</b>		<b>2391.00</b>	<b>252.70</b>	<b>447.30</b>
<b>Run # 13</b>	At Time 0, Add	700.00	20.00	2.87	0.00	700.00
<b>March 13, 2001</b>	1 <sup>st</sup> hr: 50 lb/hr			143.46	14.07	685.93
	2 <sup>nd</sup> hr: 100 lb/hr			286.92	28.15	657.78
	3 <sup>rd</sup> hr: 150 lb/hr			430.38	42.22	615.56
	4 <sup>th</sup> hr: 200 lb/hr			573.84	56.29	559.26
<b>Total Energetic Hydrolyzed</b>	<b>500.00</b>	<b>700.00</b>		<b>1434.60</b>	<b>140.74</b>	<b>559.26</b>

**Table 9-2. Demonstration Study Matrix – M28 Propellant Hydrolysis Sampling & Analysis**

Unit Operation	Validation Sampling and Analysis						
	Sampling Location (include testing purpose)	Analytes (by location and feed type)	# of Samples	Sample Collection Method	Sample Preservation Requirements	Sample Volume	Analytical Method
2,000 gal Hydrolysis Reactor System	Location: Port 3 – Figure 2  M28 Propellant Feed	a) Nitrocellulose (NC) b) Nitroglycerin (NG) c) Triacetin d) Dimethylphthalate (DMP) e) Lead Stearate f) 2-Nitro-Diphenylamine	1 Sample/Feed	Take Sample	Quench immediately in a cold (~4 °C) Sulfuric Acid	100 gram	No analysis is needed. Assume M28 Propellants is within Military Specifications
	Location: Port 5 or 6 – Figure 2  Caustic Feed NaOH Solution	a) Chloride b) Total Metals c) Carbonate	1sample/batch of NaOH feed preparation	Take Sample	N/A	a) 250 ml b) 250 ml c) 50 ml	a) SW-846-9056 b) SW-846-6010B/7470 c) ASTM E291-90
	Location: Port 4 – Figure 2  Filtered Process Water Feed	a) Chloride b) Total Metals c) Total organic Carbon (TOC)	1 Sample/Feed	Take Sample	N/A	a) 250 ml b) 150 ml c) 10 ml	a) SW-846-9056 b) SW-846-6010B/7470 c) SW-846-9060
	Location: Port 1 – Figure 2  Reactor Vessel off-gas	a) Nitrous Oxide (N <sub>2</sub> O) b) Nitrogen Oxide (NO <sub>x</sub> ) c) Nitrogen (N <sub>2</sub> ) d) Ammonia (NH <sub>3</sub> ) e) Carbon Monoxide (CO) f) Carbon Dioxide (CO <sub>2</sub> ) g) Hydrogen Cyanide (HCN) h) Aldehydes & ketones i) VOCs j) Total Hydrocarbon (TOC)	Continuous Monitoring	a) CEMS b) In-Line c) Summa d) Impingers e) CEMS f) CEMS g) Impingers h) Impingers i) Summa j)	a) N/A b) N/A c) N/A d) 4°C e) 4°C f) N/A g) N/A h) 4°C i) N/A j) 4°C	a) N/A b) N/A/ c) 10 liter d) 100 ml e) N/A f) N/A g) 100 ml h) 100 ml i) 6 liter j) 100 ml	a) Method 7E b) NOISH 6600 c) TO-15/ASTM D1946 d) Method 26/CTM-027 e) Method 0010 f) Method 3A g) Method 26/CARB 426 h) Method 0011/8315A i) TO-15 j) SW-846-9060
	Location: Port 2 – Figure 2  Reactor Vessel Liquid/Solid - Hydrolysate	a) Energetic Content b) NC c) NG d) NO <sub>2</sub> /NO <sub>3</sub> e) Cyanide by-Product f) Picric Acid – Picrate Salts (Sodium, etc.) g) Total Organic Carbon (TOC) h) Total Inorganic Carbon i) Total Metals	1 Sample/Time (min)  See Table 2 (Sampling Interval Matrix)	Take Sample via Remote Sampling System (Intersystem)	a) 4°C b) 4°C c) 4°C d) 4°C e) 4°C f) 4°C g) 4°C h) 4°C i) 4°C & pH<2	a) 50 ml b) 10 ml c) 10 ml d) 10 ml e) 50 ml f) TBD g) 10 ml h) 10 ml i) 10 ml	a) DSC b) GPC/FTIR c) SW-846-8332 / ACWA-3012 d) SW-846-9056 e) SW-846-9010B/9014 f) TBD g) SW-846-9060 h) SW-846-9060 Mod. i) SW-846-6010/7470

**Table 10. Demonstration Study Matrix – M28 Propellant/Comp B4 Explosive (86/14 wt.%) Hydrolysis Processing**

Unit Operation	Test Objective	In Support of	Feed		Test Runs	Test Run Duration - Res. Time (hr)	Supporting Data Requirements	Process Streams		Test Location
			Energetic / Caustic	Quantity (lb.)				Type	Final Destination	
2,000 gal Hydrolysis Reactor System	<ul style="list-style-type: none"> <li>Hydrolyze M28 Propellant / Comp B4 Explosive from the 115-mm, GB, M55 &amp; M56 projectile and the 115-mm, GB, M56 &amp; VX, M55 &amp; M56 Rocket &amp; Rocket Warhead in support of the scale-up and design of the Lexington Bluegrass Disposal Facility</li> <li>Characterize gas, liquid, &amp; solid process streams from the energetics hydrolysis unit for selected chemical / constituents &amp; physical parameters, and the presence / absence of energetics, energetics hydrolysis products and other hazardous and toxic compounds</li> <li>Optimize process operating parameters for the M28 propellant / Comp B4 explosive hydrolysis and obtain information applicable to completing the safety analyses, permitting, and NEPA documentation that would be required to implement base hydrolysis when production is scale-up.</li> </ul>	Lexington Bluegrass Chemical Agent Disposal Facility	M28 Propellants / Comp B or Comp B4 / 12 wt.% NaOH	See Table 10-1	1	10	<ul style="list-style-type: none"> <li>Evaluate process effect on construction materials</li> </ul>	Liquid Hydrolysate	Ship to Authorized Waste Disposal Facility or to a Technology Provider, as required by the PM	Holston Army Ammunition Plant, Holston, TN
			M28 Propellants / Comp B or Comp B4 / 20 wt.% NaOH	See Table 10-1	1	10	<ul style="list-style-type: none"> <li>Confirm expected energetics reaction time</li> </ul>	Rinse Water		
			M28 Propellants / Comp B or Comp B4 / 25 wt.% NaOH	See Table 10-1	1	10	<ul style="list-style-type: none"> <li>Optimum Process Operating Parameters (Temp., Caustic Strength, Reactor Residence Time)</li> </ul>	Decon Solution (Detergent)		
					1	10	<ul style="list-style-type: none"> <li>Ratio of caustic consumed to feed</li> <li>Temperature vs. time</li> <li>Concentration vs. time</li> <li>Destruction Efficiency (DRE)</li> <li>Utility Requirements for operation and scale-up</li> </ul>	Gas Reactor Headspace		

**Table 10-1. Comp B4 / M28 Propellant Mixture Hydrolysis Schedule and Recipe**

**Energetic Material:**            **Comp B4/Leaded M28 Mixture**

**Hydrolysis Temperature:**    **87±2 °C (for all runs)**

Day	Incremental Rate Addition, Lb/ hr	Caustic Soda Heel, gal	Caustic Soda Concentration, Wt. %	Caustic Soda Needed, lb/lb Energetic	Caustic Soda Needed, gal	Caustic Soda Remaining, gal
<b>Run # 21</b> <b>April 19, 2001</b>	At Time 0, Add	700.00	20.00	5.09	0.00	700.00
	1 <sup>st</sup> hr: 50 lb/hr			254.56	26.90	673.10
	2 <sup>nd</sup> hr: 100 lb/hr			509.12	53.81	619.29
	4 <sup>th</sup> hr: 250 lb/hr			1018.24	107.61	511.67
<b>Total Energetic Hydrolyzed</b>	<b>400.00</b>	<b>700.00</b>		<b>1781.93</b>	<b>188.33</b>	<b>511.67</b>



**Table 10-2. Demonstration Study Matrix – M28 Propellant/Comp B4 Explosive (86/14 wt.%) Hydrolysis Sampling & Analysis**

Unit Operation	Validation Sampling and Analysis						
	Sampling Location (include testing purpose)	Analytes (by location and feed type)	# of Samples	Sample Collection Method	Sample Preservation Requirements	Sample Volume	Analytical Method
2,000 gal Hydrolysis Reactor System	Location: Port 3 – Figure 2 M28 Propellant Feed	a) Nitrocellulose (NC) b) Nitroglycerin (NG) c) Triacetin d) Dimethylphthalate (DMP) e) Lead Stearate f) 2-Nitro-Diphenylamine g) RDX, HMX h) TNT i) Ca Silicate, Wax	1 Sample/Feed	Take Sample	Quench immediately in a cold (~4 °C) Sulfuric Acid	100 gram	No analysis is needed. Assume Composition is within Military Specifications
	Location: Port 5 or 6 – Figure 2 Caustic Feed NaOH Solution	a) Chloride Total Metals b) Carbonate	1sample/batch of NaOH feed preparation	Take Sample	N/A	a) 250 ml b) 250 ml c) 50 ml	a) SW-846-9056 b) SW-846-6010B/7470 c) ASTM E291-90
	Location: Port 4 – Figure 2 Filtered Process Water Feed	a) Chloride b) Total Metals c) Total organic Carbon (TOC)	1 Sample/Feed	Take Sample	N/A	a) 250 ml b) 150 ml c) 10 ml	a) SW-846-9056 b) SW-846-6010B/7470 c) SW-846-9060

**Table 10-2. Demonstration Study Matrix – M28 Propellant/Comp B4 Explosive (86/14 wt.%) Hydrolysis Sampling & Analysis  
(Continued)**

	Location: Port 1 – Figure 2 Reactor Vessel off-gas	a) Nitrous Oxide (N <sub>2</sub> O) b) Nitrogen Oxide (NO <sub>x</sub> ) c) Nitrogen (N <sub>2</sub> ) d) Ammonia (NH <sub>3</sub> ) e) Carbon Monoxide (CO) f) Carbon Dioxide (CO <sub>2</sub> ) g) Hydrogen Cyanide (HCN) h) Aldehydes & ketones i) VOCs j) Total Hydrocarbon (TOC)	Continuous Monitoring	a) CEMS b) In-Line c) Summa d) Impingers e) CEMS f) CEMS g) Impingers h) Impingers i) Summa j)	a) N/A b) N/A c) N/A d) 4°C e) 4°C f) N/A g) N/A h) 4°C i) N/A j) 4°C	a) N/A b) N/A/ c) 10 liter d) 100 ml e) N/A f) N/A g) 100 ml h) 100 ml i) 6 liter j) 100 ml	a) Method 7E b) NOISH 6600 c) TO-15/ASTM D1946 d) Method 26/CTM-027 e) Method 0010 f) Method 3A g) Method 26/CARB 426 h) Method 0011/8315A i) TO-15 j) SW-846-9060
	Location: Port 2 – Figure 2 Reactor Vessel Liquid/Solids - Hydrolysate	a) Energetic Content b) NC, RDX, HMX, TNT c) NG d) NO <sub>2</sub> /NO <sub>3</sub> e) Cyanide by-Product f) Picric Acid – Picrate Salts (Sodium, etc.) g) Total Organic Carbon (TOC) h) Total Inorganic Carbon i) Total Metals (Pb)	1 Sample/Time (min)  See Table 2 (Sampling Interval Matrix)	Take Sample via Remote Sampling System (Intersystem)	a) 4°C b) 4°C c) 4°C d) 4°C e) 4°C f) 4°C g) 4°C h) 4°C i) 4°C & pH<2	a) 50 ml b) 10 ml c) 10 ml d) 10 ml e) 50 ml f) TBD g) 10 ml h) 10 ml i) 10 ml	a) DSC b) GPC/FTIR c) SW-846-8332 / ACWA-3012 d) SW-846-9056 e) SW-846-9010B/9014 f) TBD g) SW-846-9060 h) SW-846-9060 Mod. i) SW-846-6010/7470

**Table 11. Sampling Log**

<b>Test Plan Condition #:</b>	
<b>Date Conducted:</b>	
<b>Energetic Material Tested:</b>	
<b>Reactor Operating Parameters</b>	
Hydrolysis Reactor Temperature, °C	
Reactor Residence Time, hr	
Total Energetic Material Feed, Lb.	
Alkaline Material Used	
Total Alkaline Solution Feed, Lb.	
Alkaline Concentration, wt. %	
Acid Neutralization Used	
Total Acid Feed, Lb.	
Acid Concentration, wt. %	
Total De-Ionized Water Feed, Lb.	
<b>Sampling Location</b>	<b>Analyte</b>
Reaction Vessel	RDX, HMX, TNT, Tetryl
Liquid/Solids Hydrolysate	Nitroglycerin
	Nitrocellulose
	Nitrate/Nitrite (NO <sub>2</sub> /NO <sub>3</sub> )
	Cyanide by Products
	Picric Acid – Picrate Salts (Sodium, etc.)
	Total Organic Carbon (TOC)
	Total Inorganic Carbon
	Total Metals
	pH *
Gaseous Outlet	Nitrous Oxide (N <sub>2</sub> O)
	Nitrogen Oxide (NO <sub>x</sub> )
	Nitrogen (N <sub>2</sub> )
	Ammonia (NH <sub>3</sub> )
	Carbon Monoxide (CO)
	Carbon Dioxide (CO <sub>2</sub> )
	Hydrogen Cyanide (HCN)
	Aldehydes & Ketones
	VOC
	Total Organic Carbon (TOC)
Solid Residue **	RDX, HMX, TNT, Tetryl
	Nitroglycerin
	Nitrocellulose
	Cyanide by-Product
	Picric Acid – Picrate Salts (Sodium, etc.)
	Total Organic Carbon (TOC)
	Total Inorganic Carbon
	Total Metals
	Particle Size
Energetic Destruction Efficiency (DRE) @ Residence Time Interval, %	

\* Hydrolysate pH is only measured at the end of each test condition.

\*\* Solid Residue analysis is only done at the end of each test condition. After each run residue sample will be collected from inside the reactor (bottom and sides) and analyzed for specified components.

## SYSTEM/HARDWARE PERFORMANCE EVALUATIONS

At the conclusion of the testing, the manufacturer of the reactor (Pfaudler, Inc.) will determine wear to the glass-lined vessel and the Hastelloy-C agitator to assess equipment life (wear/durability) from a material of fabrication selection standpoint. In addition, the following hardware/system assessments will be made:

- Performance of the:
  - ✓ pH sensor to operate in the hydrolysis environment
  - ✓ Acoustic level sensor
  - ✓ Intersystems Remote Sampling System
  - ✓ Process valves and pumps to handle the hydrolysate
  - ✓ Control system and its logic program
  - ✓ Flow meters/control valves for caustic, acid and water
  - ✓ Down-comer tubes (including the perforated recycle down-comer tube)
  - ✓ Internal baffling
- Efficiency of the recycle loop to maintain all solids in suspension
- Determine if:
  - ✓ Steps to control the formation of foam are effective
  - ✓ The jacket (using process water) cooling capability is sufficient or a chiller system is required for optimum performance
  - ✓ Determine if the condenser captures and returns all distillates to the vessel
- Maintenance requirements for the system (including any special needs of specific hardware)
- Safety:
  - ✓ Develop a contingency plan to respond to a sudden shut down of the system. (The concern is that the shutdown occurs during the early phase of the hydrolysis reaction when heat generation is at a maximum. Once there is a system shut down there is a need to know what's inside the reactor to make sure that it is safe to clean.)